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PROJECT FOR PERFORMANCE OF REMEDIAL RESPONSE ACTIVITIES AT UNCONTROLLED HAZARDOUS SUBSTANCE FACILITIES—ZONE 1

NUS CORPORATION
SUPERFUND DIVISION

ORIGINAL
(Red)

R-585-7-3-14

A FIELD TRIP REPORT FOR
FMC BALTIMORE
PREPARED UNDER

TDD NO. F3-8306-20
EPA NO. MD-17
CONTRACT NO. 68-01-6699

FOR THE

HAZARDOUS SITE CONTROL DIVISION
U.S. ENVIRONMENTAL PROTECTION AGENCY

FEBRUARY 22, 1984

NUS CORPORATION
SUPERFUND DIVISION

SUBMITTED BY

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APPROVED BY

GARTH GLENN
MANAGER, FIT III

ORIGINAL (Red)

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The team proceeded to Building #91, located on the north side of Patapsco Ave. The facility, formerly the pilot plant for the Tetradifon product, is currently used for "Pounce" (permethrin) production. An area of soil was observed behind the building, amidst the production plant's waste treatment area. An area of grass covered soil, containing scrub growth and small trees, was located near the building's southeast corner. An asphalt parking lot/driving area bordered the building. The area occupied by the building is bordered on three sides (north-east-west) by three different companies. The interior of the building contained Pounce-related equipment, with the piping displaying fairly recent painting.

The team proceeded to the CERCLA-fill areas located south of Building #91 and north of Patapsco Ave. The area consisted of foundations for former acetic acid production facilities. The area was very overgrown and contained standing water, due to the previous heavy rainfall of June 19, 1983. One specific area was pointed out by Mr. Palmer as an area that FMC knew contained unknown, miscellaneous material. As far as the rest of the area, Mr. Palmer had no information.

A third CERCLA area was pointed out by Mr. Palmer, located northwest of Building #91's parking lot. The area was reportedly the former location of a tank of unknown origin and contents.

Following the completion of the inspection, personnel returned to the conference room for development/discussion of the sampling plan. Upon completion of the discussion, FIT III personnel departed the site at approximately 1330 hours.

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FIT III personnel returned to the plant on June 21, 1983, to conduct sampling. Prior to the inspection, FIT III personnel were briefed on FMC safety procedures. Lines of communication, investigation protocols, and sample locations were also discussed. Personnel then proceeded to the north side of the facility (north of Patapsco Ave.). After establishment of the Command Post (CP) near Building #89, sampling was initiated.

Three bore holes were advanced on the northern, eastern, and western boundaries of the old acetic acid production area, which was reported by the plant under CERCLA. There were no signs of environmental contamination. Sample numbers M-02-01,02, and 03 were obtained.

Sampling personnel then obtained sample number M-02-04 from the bed of a railroad spur which serviced the plant. The spur was located on the plant's northeast quadrant. The material from the railroad bed consisted of a crumbly, black solid, which did not display signs of environmental contamination.

Sample number M-02-05 was obtained from the location of the storage tank, which was also reported by the plant under CERCLA. A duplicate sample, in addition to the split samples, was obtained from this station for QA/QC purposes. There was no sign of environmental contamination.

Sample numbers M-02-06 and M-02-07 were obtained from around Building #91. Sample number M-02-06 was obtained from a grass/soil area located amidst the process works for the permethrin produced at Building #91. There were no signs of obvious environmental contamination.

Sample number M-04-07 was obtained from a small lawn area located in the front of Building #91. A surface soil sample was obtained from this station. There were no signs of environmental contamination.

Sample number M-04-08 consisted of decontamination rinsate (1,1,1-trichloroethane) used to decontaminate the blender tops. This sample was also obtained in accordance with QA/QC requirements.

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Upon the completion of the processing for sample number M-02-08, the CP was transferred to the plant's southern side (south of Patapsco Ave.). Sampling on the facility's south side initiated at the inactive fill/waste pond area. Sample numbers M-02-09 and M-02-10 were obtained from the eastern and western boundaries of the area. There were no signs of environmental contamination. To obtain the samples, the top layer of ground and sand was removed, and a shallow stem auger was used to obtain the samples.

An attempt was made to sample the East Gate area (#M-02-11). However, the gravel pack was too dense and a sample could not be obtained.

The sampling personnel proceeded to the former location of Building #51, adjacent to Building #34, to obtain sample numbers M-02-12 and M-02-13. Sample M-02-12 was obtained from an area located near Building #34's southeast corner. Sample M-02-13 was obtained from soil underneath a foundation for current steps located near Building #34's northeast corner. As with sample numbers M-02-09 and M-02-10, the top layers of gravel and sand were removed, then the auger was advanced to refusal. No signs of environmental contamination were noted.

A final sample, intended as a clean field blank, was obtained from a lawn located in front of Building #19. A duplicate sample, in addition to the splits, was obtained for QA/QC purposes.

All samples for dioxin analysis were processed in accordance with Document #C-585-6-3-54 (See Appendix A). Upon completion of the sampling, split samples were provided to FMC personnel under chain-of-custody. All samples were obtained, photographed, processed, documented, packaged, and shipped in accordance with accepted protocols. All solid and liquid wastes generated during the inspection were drummed and removed from site. Upon completion of the breakdown of the CP, FIT III personnel departed the site at 1815 hours.

2.5 PHOTOGRAPHIC LOG

Photo 1-Sample location M-02-01.

Photo 2-Sample location M-02-02.

#1 Location

F3-8306. 20
6|21|83
10:50

Photo 1

Sample location M-02-01.

Terrence A. Shannon
for FMC Representative

#2 Location

F3-8306-20
6|21|83
11:20

Photo 2

Sample location M-02-02.

Terrence A. Shannon
for FMC Representative

Photo 3-Sample location M-02-03.

Photo 4-Sample location M-02-04.

#3 Location

F3-8306-20
6/21/83
11:45

Photo 3

Sample location M-02-03.

Terrence A. Shannon
for FMC Representative

#4 Location

F3-8306-20
6/21/83
12:12

Photo 4

Sample location M-02-04.

Terrence A. Shannon
for FMC Representative

Photo 5-Sample location M-02-05.

Photo 6-Sample location M-02-06.

#5 Location

Photo 5

F3-8306-20
6/21/83
12:44

JUNE 1983 JUNE 1983 JUNE 1983

Sample location M-02-05.

Terrence A. Shannon
for FMC Representative

Photo 5-Sample location M-02-05.

#6 Location

Photo 6

F3-8306-20
6/21/83
13:09

JUNE 1983 JUNE 1983 1983

Sample location M-02-06.

Terrence A. Shannon
for FMC Representative

Photo 6-Sample location M-02-06.

Photo 7-Sample location M-02-07.

Photo 8-Sample location M-02-09.

#7 Location
F3-8306-20
6/21/83
13:29

Photo 7

JUNE 1983

Sample location M-02-07.

Terrence A. Shannon
for FMC Representative

[redacted]

#9 Location
F3-8306-20
6/21/83
1536

Photo 8

JUNE 1983

Sample location M-02-09.

Terrence A. Shannon
for FMC Representative

Photo 9-Sample location M-02-10.

Photo 10-Sample location M-02-12.

#9 Location

F3-8306-20
6/21/83
15:55

Photo 9

JUNE 1983

Sample location M-02-10.

Terrence A. Shannon
for FMC Representative

#12 Location

F3-8306-20
6/21/83
16:35

Photo 10

JUNE 1983

Sample location M-02-12.

Terrence A. Shannon
for FMC Representative

Photo 11-Sample location M-02-13.

Photo 12-Sample location M-02-14.

#13 Location

F3-8306-20

6/21/83

16:51

Photo 11

Sample location M-02-13.

Terrence A. Shannon
for FMC Representative

#14 Location

F3-8306-20

6/21/83

17:15

Photo 12

Sample location M-02-14.

Terrence A. Shannon
for FMC Representative

SECTION 1

ORIGINAL (Red)

1.0 INTRODUCTION

1.1 Authorization

NUS Corporation performed this work under Environmental Protection Agency Contract No. 68-01-6699. This specific report was prepared in accordance with Technical Directive Document No. F3-8306-20 for the FMC Inc. Baltimore Plant, located in Curtis Bay, MD.

1.2 Scope Of Work

NUS FIT III was tasked to conduct sampling of FMC's Baltimore Plant in Curtis Bay, MD. Samples were analyzed for dioxin (2,3,7,8-TCDD) and priority pollutants. The investigation was conducted by NUS personnel Terrence Shannon, Eugene Dennis, Richard Cromer, Michael Nalipinski, and David Hassrick.

1.3 Summary

In accordance with the listing of FMC's Baltimore plant in the EPA "Dioxin Report", NUS FIT III conducted a sampling program consisting of screening for dioxin (2,3,7,8-TCDD) and organic and inorganic priority pollutants.

NUS FIT III personnel attended a number of meetings in preparation for the investigation. A meeting was held with personnel from the Center for Disease Control (CDC) in Atlanta, Georgia. In order to ensure a sampling program that would complement EPA III's and CDC's needs, discussions were held regarding the type of sampling plans (screening vs. statistical), sample locations (pipes, tanks, dust, etc.), quality assurance/control programs (performance audit, duplicate, and spiked samples), and on-site procedures (homogenization of samples, personnel protection).

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In addition, a meeting of the Dioxin Work Group was attended by NUS FIT III personnel, in which administrative and technical details were finalized. Prior to initiating the on-site work, briefings were held with the individual work teams to ensure that all aspects of the investigation were performed in accordance with the newly established protocols for sample preparation and sample/personnel decontamination.

EPA III officials initially contacted FMC, Inc. with a letter requesting information related to the processing and handling of the Tetradifon ("Tedion") noted in the Dioxin report. At that time, a date was arranged for both a preliminary meeting, as well as the actual sampling.

The on-site meeting and investigation took place on June 20, 1983. The preliminary meeting was attended by personnel from NUS, FMC, EPA III, the MD WRA, and the Baltimore Dept. of Health. NUS FIT III personnel initiated the sampling of those locations decided upon by EPA III at the meeting. Samples were obtained from 12 locations on the plant property.

Sample analytical results were received on 7/25/83. The results did not detect 2,3,7,8-TCDD in any of the 12 samples taken from FMC property. However, a QA/QC check of the dioxin results indicated interferences in the results for sample number M-02-13. It should be noted that only 2,3,7,8-TCDD results are presented in this report. Results of sample analysis for priority pollutants will be forthcoming in a separate report.

SECTION 2

2.0 FIELD TRIP REPORT

2.1 Summary

Pursuant to the Technical Directive Document #F3-8306-20, site sampling of the FMC, Inc., Baltimore plant was conducted on June 21, 1983. NUS FIT III personnel who participated in the inspection included Terrence Shannon, Eugene Dennis, Richard Cromer, Michael Nalipinski, and David Hassrick (Dioxin Team "B").

The weather during the inspection was overcast, with a temperature of 70° F and winds from 0-5 mph. A slight rainfall of short duration occurred during the inspection.

Samples were obtained from twelve stations at the plant for dioxin and organic/inorganic priority pollutant analyses. Split samples were provided FMC personnel, under chain-of-custody.

2.2 Persons Contacted

2.2.1 Prior to Field Trip

All contacts prior to the on-site work with the facility were made by EPA III's representative, Neil Swanson (3AW23). NUS FIT III personnel did not have any contact with facility personnel until arriving at the site.

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2.2.2 At the Site

Upon arriving at the site, a meeting was held with the following:

Peter Schaul
Neil Swanson
EPA, Region III
6th and Walnut St.
Philadelphia, PA 19106
(215) 597-9800

Darryl Palmer, Environmental Manager
Frank Soleck, Production Manager
Irv Kipnis, Process Laboratory Manager
Chuck Shaheen, Environmental Engineer
FMC Corporation
Agricultural Chemical Group
1701 E. Patapsco Ave., Box 1616
Baltimore, MD 21203
(301) 355-6400

Donald Senovich, FIT Manager
Terrence Shannon, Engineer
NUS Corporation
992 Old Eagle School Road
Wayne, PA 19087
(215) 687-9510

Joseph Stang, Inspector
MD Dept of Health & Mental Hygiene
Office of Environmental Programs
201 W. Preston St., Rm. 2A4
Baltimore, MD 21201
(301) 383-6650

Elkins Dahle, Jr., Director
Charisse Deutch, Inspector
Baltimore Bureau of Industrial Hygiene
111 N. Calvert St., Rm S-219
Baltimore, MD 21202
(301) 396-4477

TDD Number 8306-20
EPA Number MD-17

SAMPLE LOG
Dioxin

Station Number Organic	Station Number Inorganic	Station Number 2,3,7,8-TCDD High Hazard	Tag Number	PHASE/ Conc.	SAMPLE DESCRIPTION	DATE	TIME	pH		LABORATO
		M-02-01	3-15550	Solid/Low	Facility - N side	6/21/83	1040			Envirodyn
		M-02-02	3-15718		"		1111			
		M-02-03	3-14593		"		1138			
		M-02-04	3-14599		Facility - S side		1208			
		M-02-05	3-12084		Facility - N side		1237			
		M-02-06	3-12088		"		1305			
		M-02-07	3-12091		"		1326			
		M-02-08	3-12094		"		1340			
		M-02-09	3-12095		Facility - S side		1523			
		M-02-10	3-12098		"		1549			
		M-02-12	3-24054		"		1630			
		M-02-13	3-24057		"		1648			
		M-02-14	3-24060		"		1712			
		M-02-14	3-24061		"		1717			
		M-02-15	3-24901		Performance audit	6/29/83	1630			
		M-02-16	3-24902		Performance audit	6/29/83	1640			
		M-01-01	3-24064	Solid/Hi	QA Duplicate Transfer	6/21/83	1055			

ORIGINAL (Red)

ORIGINAL (Red)

2.4 Site Observations

FIT III personnel arrived on-site at 0900 on June 20, 1983, to attend a meeting previously arranged by EPA III representative Neil Swanson. Mr. Swanson described the screening nature of the anticipated sampling program and the analyses to be run (2,3,7,8-TCDD and organic/inorganic priority pollutants). A general discussion ensued, concerning the plant's layout and the production history of Tetradifon, the compound of concern to EPA due to its potential for dioxin contamination. The following information was elicited from the FMC personnel present at the meeting.

The total size of the plant is approximately 50 acres, with 20 acres located north of Patapsco Ave. and 30 acres located south of Patapsco Ave. Approximately 350 people are employed at the plant.

Relative to the production of Tetradifon, Building #91 was the location of the compound's pilot plant. Building #91 was reconfigured and is currently the site of "Pounce" (permethrin) production, a chemical used on cotton and tobacco. The equipment used for the Tetradifon production was either decontaminated and used elsewhere in the plant or scrapped and sold. The exact fate of this equipment was unknown.

The semiworks for the commercial production of the Tetradifon was located in Building #52 and was in operation from approximately 1960 through 1970. Building #52 was demolished, date unknown, and is currently the location of the plant's RCRA waste facility. This area is sealed with an asphalt pad.

All product generated by the pilot plant and the semiworks for Tetradifon were drummed and transported by truck. There was no railroad transport, to the best of the FMC personnel's recollection. Likewise, an incinerator constructed on the plant's property in 1968 was not used for the incineration of any Tetradifon production wastes.

ORIGINAL (Red)

Three CERCLA areas of the plant were discussed. One area was located adjacent to the old "waste-pond" area in the plant's southeast quarter. FMC personnel stated that the area, formerly a wetlands, was used for the disposal of aqueous waste from the production of Tetradifon, as well as unknown, miscellaneous materials. The pond was excavated, filled, and a storage facility was constructed on the site. The pond's contents were removed and possibly disposed of at Solley Road landfill. The area is currently capped with a sand/gravel cover. A second CERCLA area, located south of Building #91 and north of Patapsco Ave. was also used for the disposal of unknown, miscellaneous materials. It is the former location of acetic acid production facilities. A third CERCLA area, located approximately 200 feet west of Building #89, was the location of a tank of unknown contents and fate.

The plant has two runoff collection systems. One system, called the plant general system, drains the entire plant except for the 7-OH production area. The general system discharges to a POTW. The collection system for the 7-OH area discharges to the retention basin located on the plant's southern boundary. Effluent from the retention basin is discharged via an NPDES permit to the Patapsco River.

In regard to the technical aspects of the inspection, it was decided that splits of both the dioxin and priority pollutant samples would be provided FMC. Material for the dioxin analysis would be collected in the blender top, homogenized with the blender, and split. Material for the priority pollutant analysis would be collected in a stainless steel bucket, mixed with a stainless steel scoopula, and split. FMC would provide their own glassware for their split samples. Photographs of the sampling would be obtained by FMC, developed, and mailed to the NUS FIT III contact, Terrence Shannon. Finally, FMC personnel would accompany NUS FIT III personnel during all plant surveys and sampling.

Following the discussion, a walk-through of the facility was conducted. See next page.

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The southeast section of the plant was inspected, including the retention basin, the fire water basin, the old waste pond area (near the 7-OH control room, Building #80), and a CERCLA-reported inactive fill area. Both the old waste pond area and the CERCLA fill area were covered with what appeared to be a white, 2 to 3 inch-sized gravel, with an underlying area of fine sand, which presumably was underlain by the old waste areas. There were no signs of environmental contamination in those areas.

The inspection team proceeded to the unnamed stream which bordered the eastern portion of the south part of the facility. Recent heavy rains had severely flooded this area. Access to this portion of the facility was via the east gate, which was used at one time by all contractors entering the plant.

The inspection team proceeded to the former location of Building #52, used for production of the potential dioxin contaminated product Tetradifon. The area is currently occupied by a RCRA waste storage facility and is completely covered by asphalt and gravel of the type mentioned earlier. An area of sediment accumulation was observed underneath steps on the northwest side of the area. An open drain was observed in a shallow depression area on the southwest side of the area. A railroad spur, running north/south, was located on the other side of a plant access road located immediately adjacent to the area's western side. Building #34 was located to the west of the area.

The team proceeded to the warehouse area adjacent to the plant's 2nd St. A railroad spur, embedded in a concrete causeway, was located parallel to 2nd St., next to the warehouse. The loading platform for the warehouse area was observed at the western end of the buildings. The railroad bed material could not be ascertained.

The team proceeded to Building #91, located on the north side of Patapsco Ave. The facility, formerly the pilot plant for the Tetradifon product, is currently used for "Pounce" (permethrin) production. An area of soil was observed behind the building, amidst the production plant's waste treatment area. An area of grass covered soil, containing scrub growth and small trees, was located near the building's southeast corner. An asphalt parking lot/driving area bordered the building. The area occupied by the building is bordered on three sides (north-east-west) by three different companies. The interior of the building contained Pounce-related equipment, with the piping displaying fairly recent painting.

The team proceeded to the CERCLA-fill areas located south of Building #91 and north of Patapsco Ave. The area consisted of foundations for former acetic acid production facilities. The area was very overgrown and contained standing water, due to the previous heavy rainfall of June 19, 1983. One specific area was pointed out by Mr. Palmer as an area that FMC knew contained unknown, miscellaneous material. As far as the rest of the area, Mr. Palmer had no information.

A third CERCLA area was pointed out by Mr. Palmer, located northwest of Building #91's parking lot. The area was reportedly the former location of a tank of unknown origin and contents.

Following the completion of the inspection, personnel returned to the conference room for development/discussion of the sampling plan. Upon completion of the discussion, FIT III personnel departed the site at approximately 1330 hours.

FIT III personnel returned to the plant on June 21, 1983, to conduct sampling. Prior to the inspection, FIT III personnel were briefed on FMC safety procedures. Lines of communication, investigation protocols, and sample locations were also discussed. Personnel then proceeded to the north side of the facility (north of Patapsco Ave.). After establishment of the Command Post (CP) near Building #89, sampling was initiated.

Three bore holes were advanced on the northern, eastern, and western boundaries of the old acetic acid production area, which was reported by the plant under CERCLA. There were no signs of environmental contamination. Sample numbers M-02-01,02, and 03 were obtained.

Sampling personnel then obtained sample number M-02-04 from the bed of a railroad spur which serviced the plant. The spur was located on the plant's northeast quadrant. The material from the railroad bed consisted of a crumbly, black solid, which did not display signs of environmental contamination.

Sample number M-02-05 was obtained from the location of the storage tank, which was also reported by the plant under CERCLA. A duplicate sample, in addition to the split samples, was obtained from this station for QA/QC purposes. There was no sign of environmental contamination.

Sample numbers M-02-06 and M-02-07 were obtained from around Building #91. Sample number M-02-06 was obtained from a grass/soil area located amidst the process works for the permethrin produced at Building #91. There were no signs of obvious environmental contamination.

Sample number M-04-07 was obtained from a small lawn area located in the front of Building #91. A surface soil sample was obtained from this station. There were no signs of environmental contamination.

Sample number M-04-08 consisted of decontamination rinsate (1,1,1-trichloroethane) used to decontaminate the blender tops. This sample was also obtained in accordance with QA/QC requirements.

Upon the completion of the processing for sample number M-02-08, the CP was transferred to the plant's southern side (south of Patapsco Ave.). Sampling on the facility's south side initiated at the inactive fill/waste pond area. Sample numbers M-02-09 and M-02-10 were obtained from the eastern and western boundaries of the area. There were no signs of environmental contamination. To obtain the samples, the top layer of ground and sand was removed, and a shallow stem auger was used to obtain the samples.

An attempt was made to sample the East Gate area (#M-02-11). However, the gravel pack was too dense and a sample could not be obtained.

The sampling personnel proceeded to the former location of Building #51, adjacent to Building #34, to obtain sample numbers M-02-12 and M-02-13. Sample M-02-12 was obtained from an area located near Building #34's southeast corner. Sample M-02-13 was obtained from soil underneath a foundation for current steps located near Building #34's northeast corner. As with sample numbers M-02-09 and M-02-10, the top layers of gravel and sand were removed, then the auger was advanced to refusal. No signs of environmental contamination were noted.

A final sample, intended as a clean field blank, was obtained from a lawn located in front of Building #19. A duplicate sample, in addition to the splits, was obtained for QA/QC purposes.

All samples for dioxin analysis were processed in accordance with Document #C-585-6-3-54 (See Appendix A). Upon completion of the sampling, split samples were provided to FMC personnel under chain-of-custody. All samples were obtained, photographed, processed, documented, packaged, and shipped in accordance with accepted protocols. All solid and liquid wastes generated during the inspection were drummed and removed from site. Upon completion of the breakdown of the CP, FIT III personnel departed the site at 1815 hours.

SECTION 3

3.0 LABORATORY DATA

3.1 SAMPLE DATA SUMMARY

SAMPLE DATA SUMMARY
TARGET COMPOUNDS

TDD Number F3-8306-20
EPA Number MD-

☒ ~~Organic~~ 2,3,7,8-TCDD ☐ Inorganic

Site Name Fmc, Baltimore
Date of Sample 6-21-83

Sample Number	Sample Description and Location	Phase	Units	Compounds Detected: 2,3,7,8-TCDD	Remarks
M-02-01	Facility, North Side	SOL	ppb	ND	
M-02-02	Facility, North Side	SOL	ppb	ND	
M-02-03	Facility, North Side	SOL	ppb	ND	
M-02-04	Facility, South Side	SOL	ppb	ND	
M-02-05	Facility, North Side	SOL	ppb	ND	
M-02-06	Facility, North Side	SOL	ppb	ND	
M-02-07	Facility, North Side	SOL	ppb	ND	
M-02-08	Facility, North Side	AQ	ppb	ND	
M-02-09	Facility, South Side	SOL	ppb	ND	
M-02-10	Facility, South Side	SOL	ppb	ND	
M-02-12	Facility, South Side	SOL	ppb	ND	
M-02-13	Facility, South Side	SOL	ppb	ND	
M-02-14	Facility, South Side	SOL	ppb	ND	

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NOTE: For a review of this data and non-target, tentatively identified compounds, please see the Analytical Quality Assurance section of this report.

◇ Denotes results of questionable qualitative significance based upon quality assurance review of data.

3.2 Quality Assurance Review

3.2.1 Dioxin Data: Lab Case: SAS-619C

3.2.1.1 Introduction

The findings offered in this report are based upon a general review of all available dioxin sample data. Blank analysis, surrogate, matrix spike, duplicate, and performance audit results, calibration standards, and isomer separation standards were examined in detail.

3.2.1.2 Qualifiers

It is recommended that this data package be utilized only with a qualifier stating that the initial results for sample M-02-13 did not rule out the presence of 2,3,7,8-TCDD at the required detection limit of one part per billion. Several reanalyses of this sample were performed, and these results are addressed in Section 3.3 of this report.

3.2.1.3 Findings

- Cleanup options A and D of the Region VII protocol were used in an attempt to eliminate interferences which precluded the determination of any native 2,3,7,8-TCDD and the internal standard in sample M-02-13. The sample was re-extracted and reanalyzed in order to obtain the required detection limit, and results are discussed in a separate Quality Assurance Review (Section 3.3 of this report).

- One of the two chromatographic columns used in this project did not meet the interim isomer resolution criteria established in Kansas City on July 13, 1983. However, data generated on either column is adequate to rule out the presence of indigenous 2,3,7,8-TCDD. In order to obtain accurate quantitation and confident isomer specificity, the laboratory was directed to reinject all samples having possibly positive results for 2,3,7,8-TCDD on another column which met the criteria. (The only positive samples in this case turned out to be the two performance audit samples and the laboratory matrix spike.)

3.2.1.4 Summary

The attached Quality Assurance Review has revealed chromatographic interferences in sample M-02-13 as the major area of concern. Please see the accompanying Support Documentation Appendix to this report for specifics on this Quality Assurance Review.

Report prepared by Russell J. Sloboda Date: July 25, 1983

3.3 Quality Assurance Review

3.3.1 Reanalysis of Sample M-02-13 Dioxin Data: Lab Case: SAS-619C

3.3.1.1 Introduction

The findings offered in this report are based upon a general review of all available data for three reanalyses of sample M-02-13. The data examined represent an EPA-requested low resolution GC/MS analysis, an FMC-funded/requested high-resolution GC/MS analysis of the same sample, and an FMC-funded/requested low resolution GC/MS analysis of a split portion of this sample. (The original sample was mechanically blended in the field before splitting was performed.) EPA's low resolution GC/MS analysis detected dioxin at 1.04 ug/kg, whereas the other two analyses did not find dioxin. Detection limits were 0.27 ug/kg for the high resolution analysis and 0.20 ug/kg for the FMC low resolution analysis.

3.3.1.2 Qualifiers

It is recommended that this data package be utilized only with the following qualifier statments:

- The detection limit for the high-resolution analysis was incorrectly reported by the laboratory. The corrected limit is 0.27 ug/kg.

- Although the existing sample data is insufficient to unambiguously determine the cause of the discrepancy between the positive and non-detected sample results, several pieces of evidence suggest that the one positive result for dioxin may be an artifact of chemical interference(s) which exhibit a response similar to that of dioxin.

3.3.1.3 Findings

- The error in the reported detection limit arises out of the interpretation of the section of the dioxin protocol which addresses calculation of detection limits. The corrected limit was calculated as 2.5 times the amount represented by the lower level interfering mass areas compared to the corresponding $^{C}13_{12}$-TCDD mass area. This is different from the reported detection limit, which was calculated using the sum of the two masses 320 and 322, and which yielded a higher result due to a relatively higher interference at mass 322 versus mass 320.

- The sample contained high level interferences which necessitated additional preparatory effort for all three laboratories. Even after cleanup, multiple interferences containing co-maximizing mass 320 and 322 peaks were observed by all laboratories. In the FMC-funded low resolution GC/MS analysis, one particular interference displayed the correct 320/322 ion ratio, but without the 257 ion or retention time characteristic of dioxin. Another interference contained all three ions 320, 322, and 257, but did not exhibit the correct 320/322 ion ratio or retention time characteristic of dioxin. (In the EPA low-resolution GC/MS analysis, dioxin was identified as a peak having the correct retention time for all three ions, and a 320/322 ion ratio within the acceptable range.)

- Conversations with several chemists have revealed that interferences have occasionally produced artifactual low-resolution GC/MS results for dioxin. In this case, the high-resolution result should be given greater credibility due to the capability of this method to eliminate artifactual interferences that the low-resolution method cannot distinguish.

- To be sure, the high-resolution result does not disprove the validity of the one positive low-resolution result, since a different aliquot was taken for each analysis, and the sample might not have been as homogeneous as expected from the field blending. However, even if the positive result is not artifactual, the results from the other two analyses suggest that the average level of dioxin present is less than 1.0 ppb.

ORIGINAL
(Red)

- Thus, in order to confidently determine if the one positive result is valid or not, the original extract would have to be reanalyzed using a partial scan or high-resolution technique. However, this analysis could be successful only if significant losses of internal standard and surrogate have not occured as a result of storage and handling of the extract.

The attached Quality Assurance Review has identified an incorrect detection limit and a possible artifactual result due to chemical interference as the major areas of concern. Please see the accompanying Support Documentation Appendix to this report for specifics on this Quality Assurance Review.

Report prepared by Russell J. Sloboda Date: November 18, 1983

ORIGINAL
(Red)

APPENDIX A

ORIGINAL
(Red)

Sampling
Blending Procedure

C-585-6-3-54

Samplers take sample in 1 qt. stainless steel blender cup.

Blender cup should be filled no more than 3/4 full.

Note: Attempt to avoid placing stones in the blender cup. Samplers should also break up large clumps of soil.

Sample is then returned to blending station.

Blending procedure will commence as follows:

1 Pulse blender five (5) times.
2 Invert blender cup several times and shake.
3 Repeat this procedure six (6) times for a total of 30 pulses.
4 Allow the blender to sit for two to five minutes to allow all dust to settle.

Person who is blending removes right glove to open sample jar, glove is put back on when filling the jar.

Sample will be removed from the blender cup utilizing scoopulas which will be disposed of when the sample jar has been filled.

Right glove is removed for the capping of the jar.

Remove baggie and rubber band and place in designated receptacle.

Sample jar is decontaminated with 1,1,1-trichloroethane if visual contamination is evident.

Sample is then tagged, and processed by the site leader.

Any material remaining in blender cup is disposed of in the waste receptacle.

Blender cup is cleaned with soap and water and scrubbed with brush if necessary.

Blender cup is filled 1/4 to 1/2 full with soapy water and agitated (blended) for 30 seconds.

Cup is then rinsed with distilled water, alcohol, and 1,1,1-TCEa. Allow to drip dry.

Sample cup is ready to receive next sample.

ORIGINAL
(Red)

APPENDIX B

ORIGINAL
(Red)

FMC Corporation

Agricultural Chemical Group
1701 East Patapsco Avenue Box 1616
Baltimore Maryland 21203
(301) 355 6400

FMC

June 29, 1983

Mr. Neil Swanson
Environmental Scientist
Waste Enforcement Section
Air and Waste Management Division (3 AW 22)
United States Environmental
Protection Agency
Region III
6th and Walnut Streets
Philadelphia, PA 19106

CERTIFIED MAIL
RETURN RECEIPT REQUESTED

Re: Response to EPA Region III
Inquiry on Possible Formation
of Dioxins

Dear Mr. Swanson:

On June 20, 1983, FMC Corporation's Agricultural Chemical Group plant in Baltimore, Maryland received a letter dated June 15, 1983 from Region III of the U. S. Environmental Protection Agency, in which EPA Region III inquired as to certain information relative to the possible formation of dioxins at FMC's Baltimore plant in the course of manufacturing and handling practices over the years involving various organic chemicals. Among other things, EPA Region III's June 15, 1983 letter requests submission within ten days of receipt of the letter of a written report on the status of discussions with EPA Region III or on other action relative to the matters set forth in the letter. By the present letter, we are providing the report thus requested. In doing so, however, we do not intend to express any opinion as to the applicability of the statutory provisions referenced in the first paragraph of EPA Region III's letter or to any other statutory requirement for such a report. In response to the information requested in Attachment II of EPA Region III's letter, we are in the process of reviewing our files. We will be in a better position to respond further to this request after this review and will be in contact with you by July 10, 1983.

On Monday, June 20, 1983, a meeting was held at the Baltimore plant involving Peter Schaul and yourself from EPA Region III, myself and other FMC representatives, and representatives from your prime contractor (NUS Corporation), the City of Baltimore Health Department, and the State of Maryland Office of Environmental Programs. At that time there was an exchange of information concerning a product, Tetradifon ("Tedion"), manufacturing at FMC's Baltimore plant

ORIGINAL
(Red)

between 1957 and 1970. During the course of the meeting, there were discussions concerning the product, its related manufacturing facilities, disposal of wastes in connection with the manufacture and handling of the product and EPA Region III's letter of June 15, 1983. The meeting concluded with a brief plant tour to identify possible locations for sampling.

On the following day, June 21, 1983, the NUS Corporation representatives returned to the plant and obtained twelve (12) split spoon core samples from various locations throughout the plant site. All samples were split with our plant laboratory personnel.

It is our understanding that NUS will analyze these samples for dioxins as well as for the 129 "priority pollutants" and that the results of such analyses will be available to us in approximately four to six weeks.

If there are questions concerning this letter, or if additional information is required, please advise me (301/355-6400, Ext. 235).

Yours very truly,

D. W. Palmer
Environmental Manager

DWP:ct

cc: Elkins W. Dahle, Jr.
City of Baltimore
Health Department
Bureau of Industrial Hygiene
111 North Calvert Street
Baltimore, Maryland 21202

Art Caple -
Joseph Stang -
State of Maryland
Office of Environmental Programs
201 W. Preston Street
Baltimore, Maryland 21201

FMC Corporation

Agricultural Chemical Group
1701 East Patapsco Avenue Box 1616
Baltimore Maryland 21203
(301) 355 6400

ORIGINAL
(Red)

FMC

July 7, 1983

Mr. Neil Swanson
Environmental Scientist
Waste Enforcement Section
Air and Waste Management Division (3AW22)
United States Environmental Protection Agency
Region III
6th and Walnut Streets
Philadelphia, PA 19106

CERTIFIED MAIL
RETURN RECEIPT REQUESTED

Re: Response to EPA Region III
Inquiry on Possible Formation of Dioxins

Dear Mr. Swanson:

This letter is to confirm my July 6th telephone conversation with Peter Schaul of EPA Region III.

Because of the difficulty in attempting to locate and review information, some of which dates back twenty-five (25) years, we have requested and Mr. Shaul has agreed to an additional ten (10) days in which to respond to the request for information contained in EPA Region III's letter of June 15, 1983.

If there are any questions in this regard, please do not hesitate to contact me.

Sincerely yours,

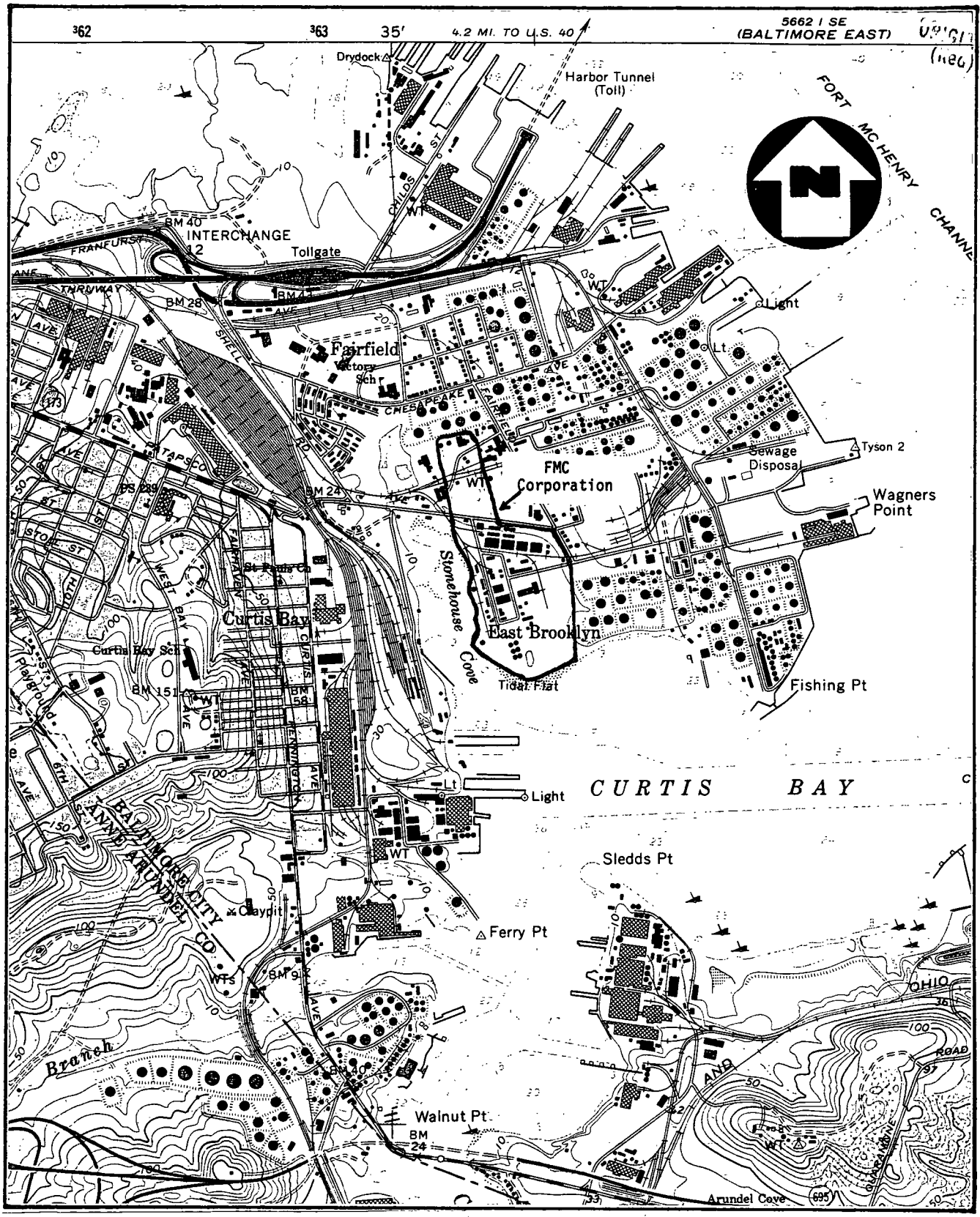
D. W. Palmer
Environmental Manager

DWP:ct

cc: Peter Schaul - EPA Region III

ORIGINAL
(Red)

APPENDIX C



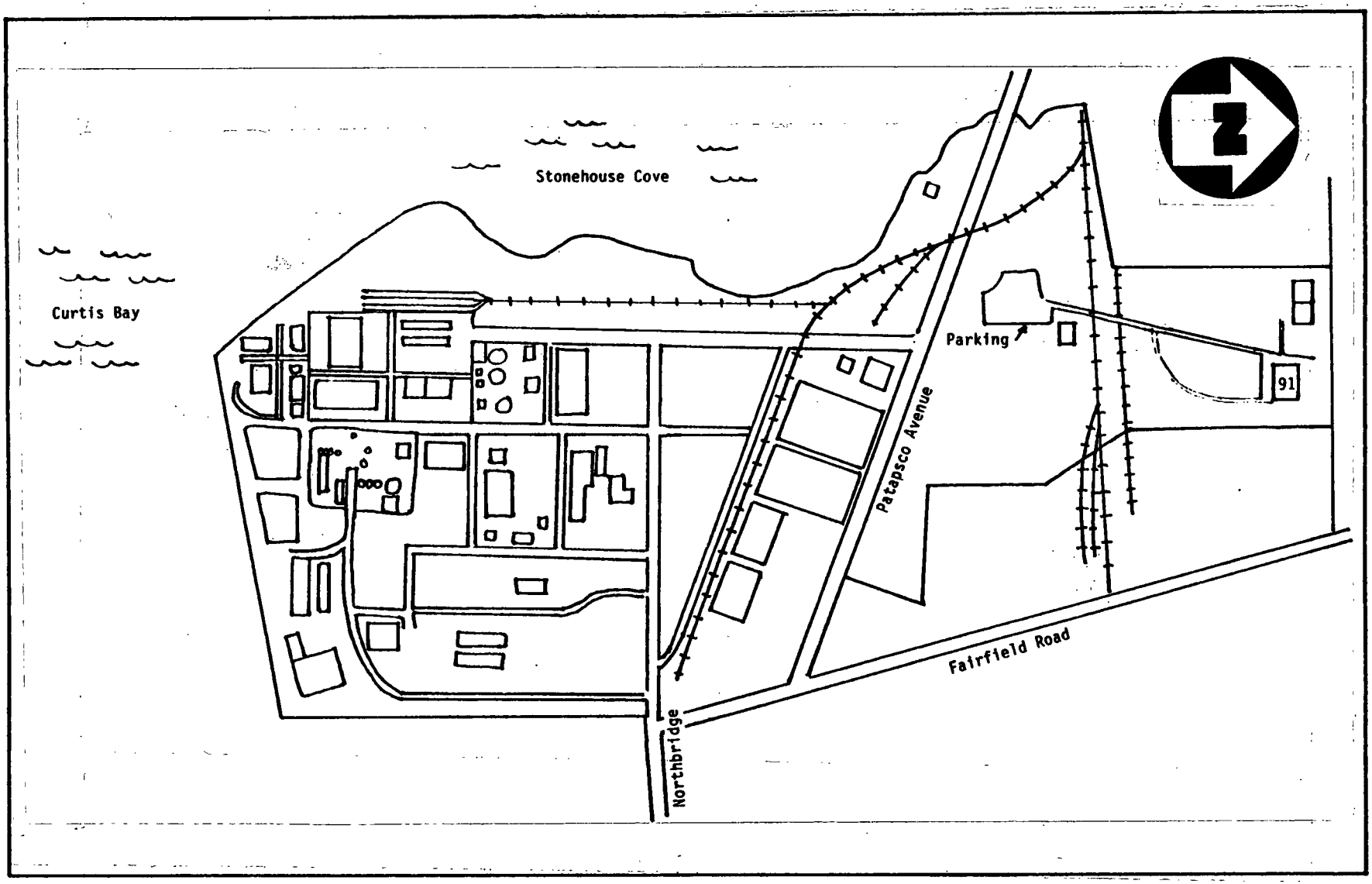
Source: USGS 7.5' Series Curtis Bay, MD Quadrangle

Site Location Map

FMC Corporation, Baltimore, Maryland

Scale: 1:24,000

NUS CORPORATION
A Halliburton Company



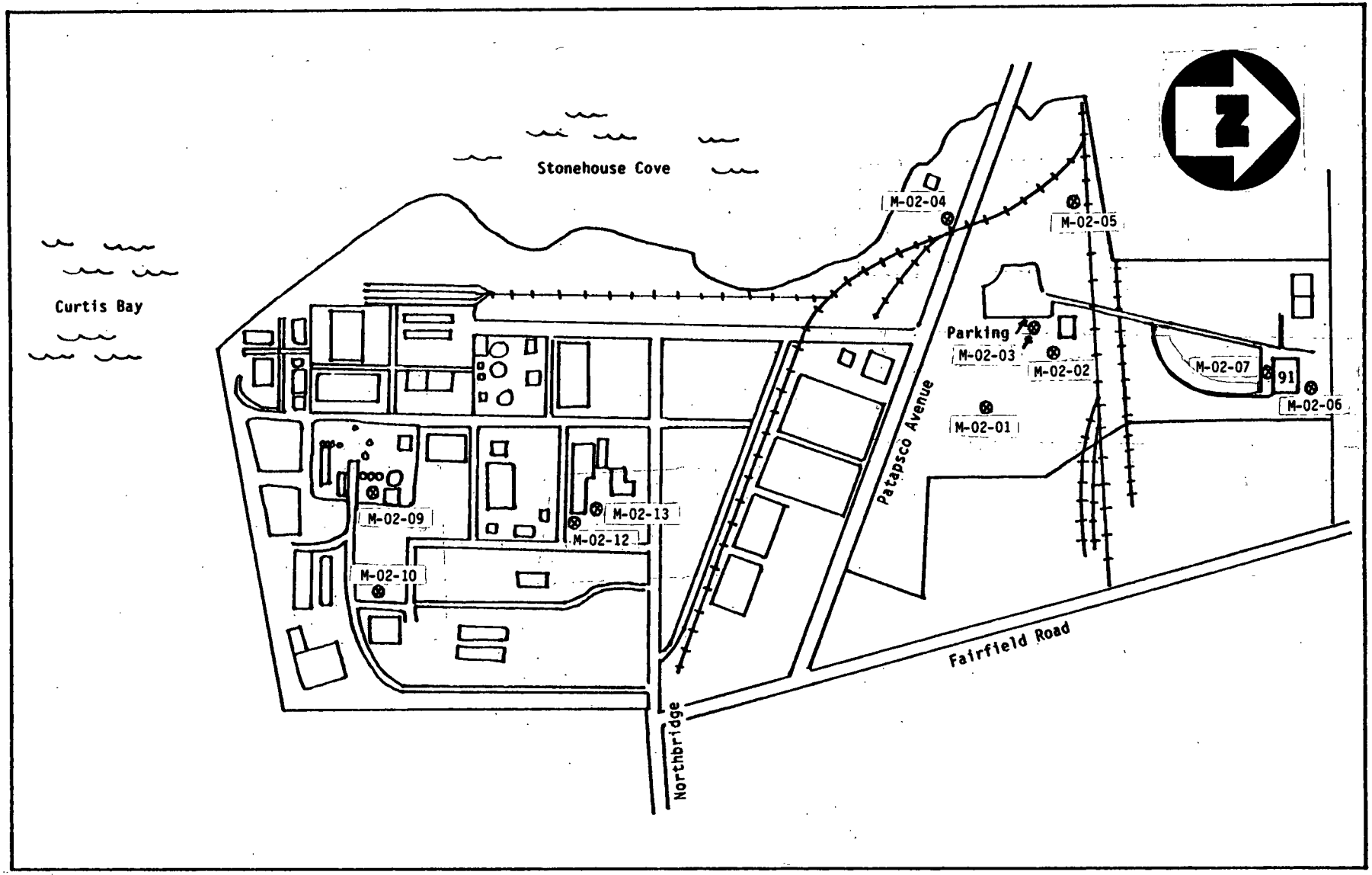
Site Sketch

FMC Corporation, Baltimore, Maryland

NOT TO SCALE

NUS CORPORATION
A Halliburton Company

ORIGINAL (Red)



Sample Location Map

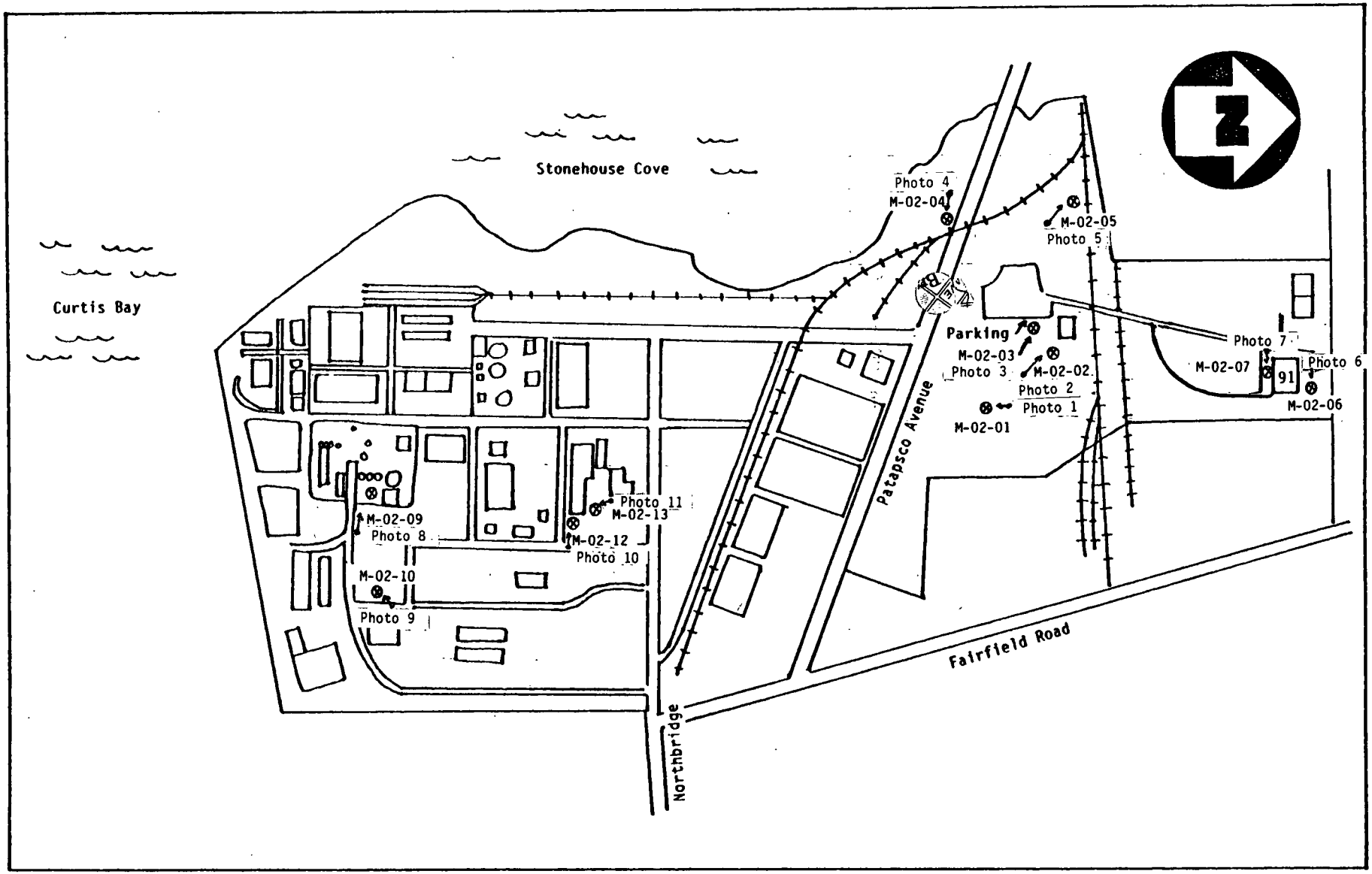
FMC Corporation, Baltimore, Maryland

NOT TO SCALE

NUS CORPORATION

A Halliburton Company

ORIGINAL (Red)



Photograph Location Sketch

FMC Corporation, Baltimore, Maryland

NOT TO SCALE

NUS CORPORATION
A Halliburton Company

ORIGINAL
(Red)

APPENDIX D

PROJECT NAME: FMC Agricultural
TDD NO: F3-8306-20

EPA SITE NO.: M-02
REGION: III

QUALITY ASSURANCE REVIEW OF DIOXIN ANALYSIS LAB DATA PACKAGE

ORIGINAL (Red)

Case No./SAS No.: 619C (Case 1836)
Contract No.: [Unknown]
Contract Laboratory: Envirodyne
Analytical Protocol: June '83 R. VII, +Memo
Reviewer: R. Slobodа
Review Date: 7/25/83

Applicable Sample No's.: M-02-01, 2, 3, 4, 5, 6, 7, 8, M-02-09, 10, 12, 13, 14, 14SPIKE, 15, 16, M-01-01

The dioxin analytical data for this case has been reviewed. The quality assurance evaluation is summarized in the following table:

Reviewer's Evaluation*	Fraction				
	2,3,7,8-TCDD	Other TCDD's	Other chlorinated dibenzodioxins	2,3,7,8-TC dibenzofuran	Other Cl'd dibenzofurans
Acceptable		‡ NA**	NA**	NA**	NA**
Acceptable with exception(s)	✓ 1				
Questionable					
Unacceptable					

* Definitions of the evaluation score categories are listed on next page.

This evaluation was based upon an analysis of the review items indicated below:

- ☑ DATA COMPLETENESS
- ☑ BLANK ANALYSIS RESULTS
- ☑ SURROGATE SPIKE RESULTS
- ☑ MATRIX SPIKE RESULTS
- ☑ DUPLICATE ANALYSIS RESULTS
- ☑ QUALITATIVE REQUIREMENTS
- ☑ CALIBRATION STANDARDS
- ☑ PERFORMANCE AUDIT RESULTS

Data review forms are attached for each of the review items indicated above.

Comments: ** Not Analyzed for. ‡ Data provided can rule out some, but not all, of the other TCDD isomers. 1 Please see surrogate spike results

DATA EVALUATION SCORE CATEGORIES

ORIGINAL
(Red)

ACCEPTABLE: Data is within established control limits, or the data which is outside established control limits does not affect the validity of the analytical results.

ACCEPTABLE WITH EXCEPTION(S): Data is not completely within established control limits. The deficiences are identified and specific data is still valid, given certain qualifications which are listed below.

QUESTIONABLE: Data is not within established control limits. The deficiences bring the validity of the entire data set into question. However, the data validity is neither proved nor disproved by the available information.

UNACCEPTABLE: Data is not within established control limits. The deficiences imply the results are not meaningful.

TCDD DATA COMPLETENESS CHECKLIST

SAMPLE NO.	M-02-01	-02-02	-02-03	-02-04	-02-05	-02-06	-02-07	-02-08	-02-09	-02-10
LAB I.D.NO. (FRN No.)	23601	23525	23526	23624	23529	23602	23623	23532	23538 (ORIGINAL) 23604	
MATRIX	soil →							solvent rinsate	soil →	
RUN DATE/TIME	7/17 8:35	7/13 14:13	7/13 15:30	7/19 13:22	7/13 17:06	7/17 5:33	7/19 14:38	7/13 18:43	7/13 11:44	7/17 16:46
INSTRUMENT I.D. NO.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
TABULATED RESULTS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
DETECTION LIMITS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
SURROGATE ACCURACY	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ION AREAS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ION RATIOS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
MID CHROMATOGRAMS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
PREVIOUS RUN AREAS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
PREVIOUS RUN CHROS.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ANALYSIS LOG	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
I. CALIB. R.F./AMTS.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
CALIB. MID.CHROS.	7/11	7/11	7/11	7/11	7/11	7/11	7/11	7/11	7/11	7/11
DAILY CALIB. RF/AMTS.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
DAILY CALIB. MID CHROS.	7/17 12:26	7/13 13:00	7/13 13:00	7/14 11:00	7/13 13:00	7/17 12:26	7/19 11:20	7/13 13:00	7/13 13:00	7/17 12:25
ISOMER SEPARATION CHROS.	7/17	7/13	7/13	7/19	7/13	7/17	7/19	7/13	7/13	7/17
STANDARD SOURCE	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
EXTRACTION WT.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
CLEANUP METHOD	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
CALCULATION VOLUMES	✓ [1] →									→
PARTIAL SCAN SPECTRA										
HIGH RESOLUTION DATA										
LAB SPIKE RECOVERY										
LAB DUPLICATE				✓						
LAB BLANK										
PERFORMANCE AUDIT SPL.										
INTER-LAB. DUPLICATE					✓*					
SAMPLE BLANK										
DECON. RINSATE								✓		

* Went to Cal. Anal. under SAS618C.

No sample M-02-11

[1] Assume final volume 50 µl, according to protocol.

TCDD DATA COMPLETENESS CHECKLIST

SAMPLE NO.	M-02-12	-02-13	-02-14	-02-14N	-02-15	-02-16	M-01-01	M-02-04D	MB LANK (Soils)	MB LANK (Powder)
LAB I.D.NO. (FRN No.)	23620	23621	23622	23539 (8912)	23541 (6931)	23542 (6918)	23547	23625	UNKNOWN 23540	UNKNOWN 23548
MATRIX	soil	→	→	→	→	→	powder	soil	unknown	unknown
RUN DATE/TIME	7/19 11:40	7/19 13:01	7/19 14:00	7/19 21:29 7/13 22:33	7/19 21:06 7/13 23:30	7/19 22:11 7/13 24:00	7/14 12:53	7/19 16:05	7/14 11:45	7/13 13:30
INSTRUMENT I.D. NO.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
TABULATED RESULTS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
DETECTION LIMITS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
SURROGATE ACCURACY	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ION AREAS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ION RATIOS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
MID CHROMATOGRAMS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
PREVIOUS RUN AREAS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
PREVIOUS RUN CHROS.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ANALYSIS LOG	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
INIT. CALIB. R.F./AMTS.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
INIT. CALIB. MID.CHROS.	7/11	7/11	7/11	7/11 / 7/19	7/11 / 7/19	7/11 / 7/19	7/11	7/11	7/11	7/11
DAILY CALIB. RF/AMTS.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
DAILY CALIB. MID CHROS.	7/19 11:16	7/19 11:00	7/19 11:00	7/19 13:00 7/13 21:23	7/19 13:50 7/13 21:13	7/19 13:56 7/13 21:23	7/14	7/19 11:00	7/14	7/13 13:00
ISOMER SEPARATION CHROS.	7/19	7/19	7/19	7/19 7/13	7/19 7/13	7/19 7/13	7/14	7/19	7/14	7/13
STANDARD SOURCE	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
EXTRACTION WT.	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
CLEANUP METHOD	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
CALCULATION VOLUMES	✓ 1	✓ 1	✓ 1	✓ 1	✓ 1	✓ 1	✓ 1	✓ 1	✓ 1	✓ 1
PARTIAL SCAN SPECTRA					✓					
HIGH RESOLUTION DATA										
LAB SPIKE RECOVERY				✓						
LAB DUPLICATE								✓		
LAB BLANK									✓	✓
PERFORMANCE AUDIT SPL.					✓	✓				
INTER-LAB. DUPLICATE							✓ ‡			
SAMPLE BLANK			✓							
DECON. RINSATE										

‡ Went to Cal. Anal. under SAS 618C.

1 Assume final volume 50 µl, according to protocol.

Blank Analysis Results

The contaminants found in the blanks are listed below:

ORIGINAL (Red)

FRACTION	TYPE OF BLANK	SAMPLE NO.	SOURCE OF water/soil	CONTAMINANTS (concentration/DL)
Soil	Sample soil Blank	M-02-14	clean soil	TCDD (ND/DL 0.46 ug/kg)
Soil	lab soil blank	MB(soils) FRN23545	Lab	TCDD (ND/DL 0.24 ug/kg)
powder	lab powder blank	MB(Powder) FRN23548	Lab	TCDD (ND/DL 0.84 ug/kg)
Rinsate	1,1,1-trichloroethane rinsate used during sampling equipment decontamination	M-02-08	1,1,1-trichloroethane	TCDD (ND/DL 0.039 ug/kg rinsate)

COMMENTS: No positive sample results from real samples.

SURROGATE SPIKE RECOVERIES

* Asterisked values are outside of QC limits

ORIGINAL (Red)

Surrogate compound name:		$^{37}Cl_4$-2,3,7,8-TCDD												
Analytical Fraction:		TCDD												
QC LIMITS:	water:													
	soil:													
	source:													
Matrix	Sample no.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.	Ref.
Soil	M-02-01	111												
"	-02	93												
"	-03	83												
"	-04	102												
"	-04D	101												
"	-05	85												
"	-06	95												
"	-07	91												
Effluent	-08	95												
Soil	-09	92												
"	-10	98												
	[No sample M-02-11]													
Soil	-12	93												
"	-13	0*												
"	-14	104												
"	-14N	93												
"	-14N[a]	104												
"	-15	94												
"	-15[a]	113												
"	-16	95												
"	-16[a]	113												
"	Method Blank (soils)	83												
Powder	M-01-01	99												
"	method Blank (powder)	93												

Source of QC Limits: Ref. 1:
Ref. 2: Instructional Guide for Reviewing GC/MS Data, version (11/5/82)

COMMENTS: *Lab note indicates interferences; is being re-extracted and reanalyzed.
The results for M-02-013 indicate that the detection limits for 2378-TCDD may be significantly above those required (1ppb). (Options A and B were used on M02-13 but the internal standard and 2,3,7,8-TCDD ions still had interferences after both cleanups.)

Matrix Spike Results (spiked by laboratory)

ORIGINAL (Red)

compound	original sample no.	spiked sample no	concentration ADDED	concentration RECOVERED	Percent Recovery	Laboratory Control Limits
2,3,7,8-TCDD	M-02-14	M-02-14N	1.0 ug/kg	1.2 ug/kg	120%	Not specified
2,3,7,8-TCDD	M-02-14	M-02-14N[a]	1.0 ug/kg	1.3 ug/kg	130%	Not specified

* An asterisk indicates values outside control limits.

Comments: Acceptable recoveries, considering that no positive results were encountered for real samples in this project.

Duplicate Analysis Results

compound	Type of duplicate (Inter/Intra-Lab)	Sample No. /Lab Name	Concentration	Sample No. /Lab Name	Conc.	Relative Percent Difference
$^{37}Cl_4$-2,3,7,8-TCDD	intra-lab	m02-04/ enviredyne	102% R	m-02-04D/ enviredyne	101% R	1%
2,3,7,8-TCDD	intra-lab	m02-04 enviredyne	ND/DL .21 ug/kg	m-02-04D/ enviredyne	ND/DL .14 ug/kg	0
2,3,7,8-TCDD	interlab	m-02-05/ enviredyne	ND/DL 0.06 ug/kg	m-02-05/ Cal Anal	Not yet available	
2,3,7,8-TCDD	interlab	m-01-01/ enviredyne	ND/DL 0.20 ug/kg	m-01-01/ Cal Anal.	Not yet available	

Control limits: Not established. Source of QC Limits: _____

* An asterisk indicates outliers.

Comments: Results not yet available for m0205 and m0101 analyses at California Analytical Laboratories.

Qualitative Requirements

ORIGINAL (Red)

A. 1. Isomer Specificity Demonstrated in Documentation? (Y/N) Yes

2. Isomer Specificity Demonstrated in Documentation within 8 hours to all positive sample runs? (Y/N) Yes; however,
Exceptions: Negative results were run only on column which did not separate isomers quite as well, but since all positives re-run on good column, data valid.

B. 1. 320/322 Ion Ratio within QC Limits (.67 - .87) for all positives?
Y/N Yes Exceptions None. Only positives were QC samples.

C. 1. 320, 322, 257 All maximize together (within 3 sec.)? (Y/N) Yes
Exceptions None

2. S/N greater than 2.5 for each ion? (Y/N) Yes Exceptions: None

D. Retention time of surrogates and internal standard same as native TCDD?
(Y/N) Yes Exceptions None

E. Confirmation Data

1. At least one confirmed per set of 24? (Y/N) Yes Exceptions None

2. High resolution confirmation? (Y/N) No Comments Partial scan good enough.

3. Partial scan confirmation? (Y/N) Yes

→ Ion Ratios:	QC Limits:	Sample:
320/322	.67-.87	0.82
320/324	1.58 ± 0.16 (1.42-1.74)	1.80 *
257/259	1.03 ± 0.10	0.95
194/196	1.54 ± 0.15	1.45

160, 161, 194, 196, 257, 259, 320, 322, 324 All present except mass 160.*
Comments Only outliers are denoted with an asterisk above. The partial scan confirmation is of confident matching quality.

Calibration Standards

Calibration data provided for 3 concentration levels? (Y/N) Yes
Exceptions: None

ORIGINAL (Red)

Linearity verified within working range? (RRF<10% RSD) Yes
Exceptions: None

Calibration Check data provided for all sample runs? (Y/N) Yes
Exceptions: None

Check standard RRF's within ±10% of multilevel calibrations? (Y/N) Yes
Exceptions: None

Average RRF from calibration used in all calculations? (Y/N) Yes
Exceptions: None

Performance Audit Results

Source of performance audit samples: Region ~~VII~~ III EPA soil; blended by Dr. Kiwe, U. of Nevada, for EMSL-LV
Date prepared: 6/28/83 Shelf Life (If applicable): NA Matrix: Soil
Interferents Added: None

Reference Analysis: None available at this time. Region III rough guess approximately 2 ppb.

Compound							
mean value							
number of measurements							
standard deviation							

Performance Audit Sample Results:

			(Reruns)						
Sample no. :	M-02-15	M-02-16	M-02-15	M-02-16					
compound :	2378	2378	2378	2378					
concentration : (ug/kg)	3.3	3.1	3.6	3.4					
mean value of audit pair (this batch):	3.35	—	—	—					
this lab's preceeding mean (last batch):	NE*	NE*	NE*	NE*					
(1.96σ) control Limits for mean (this batch):	NE*	NE*	NE*	NE*					
(2.58σ) control Limits for consecutive outliers:	NE*	NE*	NE*	NE*					
relative percent difference (RPD) for (this batch) audit pair:	8.7%	—	9.2%	—					
RPD for this lab's last batch:	NE*	NE*	NE*	NE*					
(1.96σ) control limits for RPD (this batch):	NE*	NE*	NE*	NE*					
(2.58σ) control limits for RPD consecutive:	NE*	NE*	NE*	NE*					

*An asterisk indicates values beyond 1.96 standard deviations from the mean.
**A double asterisk indicates values beyond 2.58 standard deviations from the mean.

Comments: NE* = Not yet established. This result series was the first received for this batch of performance audit material from Region III. Since no positive results were encountered in the real samples, and since performance audit samples were positively detected (and expected to be near detection limit), one can therefore assume valid method performance when all other indicators (such as surrogate recoveries) are in control.

PROJECT NAME: FMC Agricultural
TDD NO: F3-8306-20

EPA SITE NO.: M-02
REGION: III

QUALITY ASSURANCE REVIEW OF DIOXIN ANALYSIS LAB DATA PACKAGE

ORIGINAL (Red)

Case No./SAS No.: 1836/SAS 619C
Applicable Sample No's.: M-02-13 (Reanalysis)
Contract No.: [Unknown]
Contract Laboratory: Envirodyne
Analytical Protocol: July '83 RVIII + memo
Reviewer: R. Slobada
Review Date: 10/25/83

The dioxin analytical data for this case has been reviewed. The quality assurance evaluation is summarized in the following table:

Reviewer's Evaluation*	Fraction				
	2,3,7,8-TCDD	Other TCDD's	Other chlorinated dibenzodioxins	2,3,7,8-TC dibenzofuran	Other Cl'd dibenzofurans
Acceptable		Not analyzed			
Acceptable with exception(s)	✓1				
Questionable					
Unacceptable					

* Definitions of the evaluation score categories are listed on next page.

This evaluation was based upon an analysis of the review items indicated below:

- ☑ DATA COMPLETENESS
- ☑ BLANK ANALYSIS RESULTS
- ☑ SURROGATE SPIKE RESULTS
- ☑ MATRIX SPIKE RESULTS
- ☑ DUPLICATE ANALYSIS RESULTS
- ☑ QUALITATIVE REQUIREMENTS
- ☑ CALIBRATION STANDARDS
- ○ PERFORMANCE AUDIT RESULTS
- ☑ CALCULATION CHECKS

Data review forms are attached for each of the review items indicated above.

Comments: 1 Please see qualitative requirements. Only one sample from site investigation reported positive. Although this sample result suggests the presence of TCDD's, the following evidence suggests that TCDD result may be artifactual: (1) 2 other labs got a negative TCDD result, with detection limits of 0.2 and 0.27 ppb. One lab was a high resolution lab. (2.) Calculating 320 and 322 interferences, and isolated 257 interferences were seen in all analyses. The 320/322 ratio was correct for one of the peaks in the mixed analysis which was separated from the TCDD retention window. (3) Conversations with Region III Epi chemist Angelo Carasea, Cal. Analytical chemist/President Paul Taylor, and Battelle Hi-resolution GC/MS chemist Dr. Fred De Roos indicate that artifactual TCDD results do occasionally occur, although identity of the interferent cannot be determined except perhaps by full or partial scan reanalysis of the original positive extract after reconstitution and extreme concentration.

DATA EVALUATION SCORE CATEGORIES

ORIGINAL
(Red)

ACCEPTABLE: Data is within established control limits, or the data which is outside established control limits does not affect the validity of the analytical results.

ACCEPTABLE WITH EXCEPTION(S): Data is not completely within established control limits. The deficiences are identified and specific data is still valid, given certain qualifications which are listed below.

QUESTIONABLE: Data is not within established control limits. The deficiences bring the validity of the entire data set into question. However, the data validity is neither proved nor disproved by the available information.

UNACCEPTABLE: Data is not within established control limits. The deficiences imply the results are not meaningful.

TCDD DATA COMPLETENESS CHECKLIST

SAMPLE NO.	WC213											
LAB I.D. NO. (FRN No.)	7384											
MATRIX	Soil								ORIGINAL			
RUN DATE/TIME	3/26 06:46								(Red)			
INSTRUMENT I.D. NO.												
TABULATED RESULTS	✓											
DETECTION LIMITS	✓											
SURROGATE ACCURACY	✓											
ION AREAS	✓											
ION RATIOS	✓											
MID CHROMATOGRAMS	✓											
PREVIOUS RUN AREAS	ms*											
PREVIOUS RUN CHROS.	ms*											
REANALYSIS LOG												
3 PT. CALIB. R.F./AMTS.	✓											
3 PT. CALIB. MID. CHROS.	✓											
DAILY CALIB. RF/AMTS.	✓											
DAILY CALIB. MID CHROS.	✓											
ISOMER SEPARATION CHROS.	✓											
STANDARD SOURCE	✓											
EXTRACTION WT.	✓											
CLEANUP METHOD	✓											
CALCULATION VOLUMES	✓											
PARTIAL SCAN SPECTRA												
HIGH RESOLUTION DATA												
LAB SPIKE RECOVERY →	Not Performed. Data not included in package but Region III batch result given verbally											
LAB DUPLICATE →	Not Performed.	"	"	"	"	"	"	"	"	"	"	"
LAB BLANK →	Region III batch -- data not included in package but provided verbally											
PERFORMANCE AUDIT SPL.	Not analyzed with this project samples.											
INTER-LAB. DUPLICATE	"	"	"	"	"	"						
SAMPLE BLANK	"	"	"	"	"	"						
DECON. RINSATE	"	"	"	"	"	"						

ms* = conversation w/ Earl Hansen on 10/3/83 indicates previous run was ND w/ DL = 0.21.

Blank Analysis Results

The contaminants found in the blanks are listed below:

ORIGINAL (Red)

FRACTION	TYPE OF BLANK	SAMPLE NO.	SOURCE OF water/soil	CONTAMINANTS (concentration/DL)
TCDD	Lab Blank for Region VII sample	?		ND (DL = 0.08 ug/kg)

COMMENTS: Sample was run along with a region VII rerun shipment. QC data for duplicates, spikes, and method blank provided verbally in a telephone conversation w/ Dr. Earl Hanson on 10/3/83.

(Previous run ND w/ DL = 0.21 ppb per conversation above.)

SURROGATE SPIKE RECOVERIES (RELATIVE TO INT. STD.)

* Asterisked values are outside of QC limits

ORIGINAL (Red)

Surrogate compound name:		$^{37}Cl_4$-2378-TCDD			
Analytical Fraction:		TCDD			
QC LIMITS FOR SOILS	Laboratory C.L.:				
	EPA Action:	60-140			
	source:	Ref. 1	Ref.	Ref.	Ref.
QC LIMITS FOR WATER	Laboratory C.L.:				
	EPA ACTION:				
	source:	Ref.	Ref.	Ref.	Ref.
Matrix	Sample no.				
soil	mo2-13	80%			

Matrix	Sample no.					
Soils source:		Ref.	Ref.	Ref.	Ref.	Ref.
Water source:		Ref.	Ref.	Ref.	Ref.	Ref.

Source of QC Limits: Ref. 1: September '83 R. VII protocol
Ref. 2:

COMMENTS: Acceptable recovery

Matrix Spike Results (spiked by laboratory)

compound	original sample no	spiked sample no	concentration ADDED	concentration FOUND	concentration UNSPIKED	RELATIVE RECOVERY	LABORATORY CONTROL LIMITS	EPA CONTROL LIMITS	
2,3,7,8-TCDD	?	?	1.0 ppb						ORIGINAL (Red)

* An asterisk indicates values outside control limits.

Comments: 10/3 Conversation w/ Dr. Earl Hanson: Mean of 2 spikes was 72% Relative Recovery. Sample was run along with a region VIII shipment.

Duplicate Analysis Results

compound	Type of duplicate (Inter/Intra-Lab)	Sample No. / Lab Name	Concentration	Sample No. / Lab Name	Conc.	Relative Percent Difference
2,3,7,8-TCDD	inter-lab	mo2-13/ Envirodyne	1.04 ppb	mo2-13/ Battelle	ND/DL 0.13 ppb	200 %
2,3,7,8-TCDD	inter-lab	split of mo2-13/ Mead	ND/DL 0.20 ppb	mo2-13/ Envirodyne	1.04 ppb	200 %
2,3,7,8-TCDD	intra-lab	split of mo2-13/ MEAD	ND/DL 0.2 ppb	split of mo2-13/ MEAD	ND/DL 0.2 ppb	0 %

Control limits: _____ Source of QC Limits: _____

* An asterisk indicates outliers.

Comments: (Region VII samples run in Envirodyne sample batch. QC samples run in duplicate, and 5.8 and 5.9 ppb found in these analyses.)

Envirodyne was only lab to obtain a positive result for this sample.

Battelle sample was same sample as Envirodyne, EPA Sample # mo2-13. MEAD's sample was a split of the same sample, but sample was homogenized before splitting.

ORIGINAL (Red)

Qualitative Requirements

A. 1. Isomer Specificity Demonstrated in Documentation? (Y/N) Yes

2. Isomer Specificity Demonstrated in Documentation within 8 hours to all positive sample runs? (Y/N) Yes

Exceptions: None

B. 1. 320/322 Ion Ratio within QC Limits (.67 - .87) for all positives?

Y/N No Exceptions 0.66923 is slightly below 0.67. However, positive results have been seen before which are near the borderline of QC criteria.

C. 1. 320, 322, 257 All maximize together (within 3 seconds)? (Y/N) Yes

Exceptions None

2. S/N greater than 2.5 for each ion? (Y/N) Yes Exceptions: None

D. Retention time of surrogates and ~~internal standard~~ same as native TCDD? (Y/N) Yes Exceptions None

E. Confirmation Data

1. At least one confirmed per set of 24? (Y/N) No Exceptions

2. High resolution confirmation? (Y/N) Yes Comments High resolution results indicate TCDD not present with a detection limit of 0.27 ppb. However, a separate extraction was performed, so if sample not homogeneous this does not invalidate result for low resolution.

3. Partial scan confirmation? (Y/N) No

→ Ion Ratios: QC Limits:

320/322 _____

320/324 _____

257/259 _____

194/196 _____

160, 161, 194, 196, 257, 259, 320, 322, 324 _____

Comments Only one positive reported for the site investigation. Although no reason has been established the same sample analyzed by Hi-Resolution did not show TCDD. In addition, a split sample did not show the presence of dioxin with a detection limit of 0.2 ppb at another lab. (The sample was blended in the field before splitting.) Since 320 and 322 interferences were seen, it is possible that chromatographic interferences produced an artifactual result.

Calibration Standards

Calibration data provided for 3 concentration levels? (Y/N) Yes ORIGINAL (Red)
Exceptions: None

Linearity verified within working range? (RRF<10% RSD) Yes (6.9% RSD)
Exceptions: None

Calibration Check data provided for all sample runs? (Y/N) Yes
Exceptions: None

Check standard RRF's within ±10% of multilevel calibrations? (Y/N) Not applicable
Exceptions: multilevel standard within 8 hours previous to sample run.

Average RRF from calibration used in all calculations? (Y/N) Yes
Exceptions: None

CALIBRATION LOG

EQUIVALENT PPB LEVEL OF TCDD	INSTRUMENT IDENTIFIER	RUN FILE IDENTIFIER	DATE/TIME OF INJECTION	RESPONSE FACTORS: 2,3,7,8-TCDD	$^{37}Cl_4$-2,3,7,8-TCDD	ISOMER STD. CHECK STD or MULTILEVEL
25		7377	8/25 17:35	0.78	1.25	multi-level
5		7378	8/25 18:11	0.80	1.31	" "
1		7379	8/25 18:53	0.89	1.28	" "
		7380	8/25 19:26	<25% Valley		Isomer Std.

Calculation check for positive results:

MO2-13: $\frac{(87+130)}{(308+328)} \times \frac{25}{10.4} \times \frac{1}{0.8233} = 1.04$ ppb ✓

Low level Standard: FRN 7379: $RF = \frac{(1076+1388)}{(3084+3540)} \times \frac{25}{10} = 0.8897$ ✓

PROJECT NAME: FMC Agricultural
TDD NO: F3-8356-20

EPA SITE NO.: M-02
REGION: III

ORIGINAL (Red)

QUALITY ASSURANCE REVIEW OF GC/MS HIGH RESOLUTION DIOXIN ANALYSIS LAB DATA PACKAGE

Case No./SAS No.: Not EPA funded
Contract No.: " " "
Contract Laboratory: Battelle
Analytical Protocol: EPA R VII / Hi-Rez. Adapted
Reviewer: R. Slobodin
Review Date:

Applicable Sample No's.: 13,228 [FMC Split], M-02-13, M-02-14, M-02-16, M-02-14N

The dioxin analytical data for this case has been reviewed. The quality assurance evaluation is summarized in the following table:

Reviewer's Evaluation*	Fraction				
	2,3,7,8-TCDD	Other TCDD's	Other chlorinated dibenzodioxins	2,3,7,8-TC dibenzofuran	Other Cl'd dibenzofurans
Acceptable		Not analyzed for →			
Acceptable with exception(s)	✓1				
Questionable					
Unacceptable					

* Definitions of the evaluation score categories are listed on next page.

This evaluation was based upon an analysis of the review items indicated below:

- ✓ DATA COMPLETENESS
- ✓ BLANK ANALYSIS RESULTS
- ✓ SURROGATE SPIKE RESULTS
- ✓ MATRIX SPIKE RESULTS
- ✓ DUPLICATE ANALYSIS RESULTS
- ✓ QUALITATIVE REQUIREMENTS
- ✓ CALIBRATION STANDARDS
- ✓ PERFORMANCE AUDIT RESULTS
- ✓ CALCULATION CHECKS

Data review forms are attached for each of the review items indicated above.

Comments: 1 Please see calculation checks. Detection Limit for M-02-13 can be more accurately stated as 0.27 ppb. Results suggest Envirodyne results may be artifactual, or that sample was inhomogeneous. Split sample was field homogenized before being split from M-02-13. (M-02-13 sample sent to this lab (Battelle) from Envirodyne, & was blended in the field, too.)

DATA EVALUATION SCORE CATEGORIES

ORIGINAL
(Red)

ACCEPTABLE: Data is within established control limits, or the data which is outside established control limits does not affect the validity of the analytical results.

ACCEPTABLE WITH EXCEPTION(S): Data is not completely within established control limits. The deficiences are identified and specific data is still valid, given certain qualifications which are listed below.

QUESTIONABLE: Data is not within established control limits. The deficiences bring the validity of the entire data set into question. However, the data validity is neither proved nor disproved by the available information.

UNACCEPTABLE: Data is not within established control limits. The deficiences imply the results are not meaningful.

TCDD DATA COMPLETENESS CHECKLIST

SAMPLE NO.	13228	MO2-13	MO2-14	MO2-1C	MO2-14N	MB	MO2-13			ORIGINAL (Red)
LAB I.D. NO.	—	—	219907	219911	219908	219910	219913			
MATRIX	Soil →									
RUN DATE/TIME	*	*	10/19	10/19	10/19	10/19	10/19			
INSTRUMENT I.D. NO.	*	*	VG 7070H → HRMS Resolution ≥ 30 mmu at 300 amu						*	*
TABULATED RESULTS	*	*	✓	✓	✓	✓	✓			
DETECTION LIMITS	*	*	✓	✓	✓	✓	✓			
SURROGATE ACCURACY	✓	*	✓	✓	✓	✓	✓			
ION AREAS	*	*	✓	✓	✓	✓	✓			
ION RATIOS	*	*	✓	✓	✓	✓	✓			
MID CHROMATOGRAMS	*	*	✓	✓	✓	✓	✓			
PREVIOUS RUN AREAS										
PREVIOUS RUN CHROS.										
REANALYSIS LOG										
3 PT. CALIB. R.F./AMTS.			✓	✓	✓	✓	✓			
3 PT. CALIB. MID CHROS.			✓	✓	✓	✓	✓			
DAILY CALIB. RF/AMTS.			✓	✓	✓	✓	✓			
DAILY CALIB. MID CHROS.			✓	✓	✓	✓	✓ →		219915	
ISOMER SEPARATION CHROS.			✓	✓	✓	✓	✓ →		219916	
STANDARD SOURCE			✓	✓	✓	✓	✓			
EXTRACTION WT.	10.8	10.4	10.9	10.4	10.7	10.0	1.0			
CLEANUP METHOD	✓	✓	✓	✓	✓	✓	✓			
CALCULATION VOLUMES	✓	✓	✓	✓	✓	✓	✓			
PARTIAL SCAN SPECTRA										
HIGH RESOLUTION DATA	✓	✓	✓	✓	✓	✓	✓			
LAB SPIKE RECOVERY					✓					
LAB DUPLICATE										
LAB BLANK						✓				
PERFORMANCE AUDIT SPL.				✓						
INTER-LAB. DUPLICATE										
SAMPLE BLANK										
DECON. RINSATE										

* = Unable to analyze due to precipitate, so 1.0g reextracted

** per telephone conversation w/ Dr. DeRoos on 11/14/83.

Blank Analysis Results

ORIGINAL
(Red)

The contaminants found in the blanks are listed below:

FRACTION	TYPE OF BLANK	SAMPLE NO.	SOURCE OF water/soil	CONTAMINANTS (concentration/DL)
2,3,7,8-TCDD	Lab method Blank	MB	Lab	2,3,7,8-TCDD (0.01 ug/kg)

COMMENTS: Lab claims contamination due to 1/2 % carryover from previous run. (However, previous run data was not included in package) No effect on validity of data.

SURROGATE SPIKE RECOVERIES (RELATIVE TO INT. STD.)

* Asterisked values are outside of QC limits

ORIGINAL (Red)

Surrogate compound name:	$^{37}Cl_4$-2,3,7,8-TCDD										
Analytical Fraction:	TCDD										
QC LIMITS FOR SOILS — Laboratory C.L.*											
QC LIMITS FOR SOILS — EPA Action:	60-140										
QC LIMITS FOR SOILS — source:	Ref. 1	Ref.	Ref.	Ref.		Ref.	Ref.	Ref.	Ref.	Ref.	
QC LIMITS FOR WATER — Laboratory C.L.*											
QC LIMITS FOR WATER — EPA ACTION:											
QC LIMITS FOR WATER — source:	Ref.	Ref.	Ref.	Ref.		Ref.	Ref.	Ref.	Ref.	Ref.	
Matrix / Sample no.					Matrix / Sample no.						
soil	M-02-14	120									
	M-02-16	121									
	M-02-14N	119									
	M-02-13	120									
	Method Blank	119									

Source of QC Limits: Ref. 1: September '83 Revision of Region VII dioxin protocol
Ref. 2:

COMMENTS: Acceptable relative recovery.

Matrix Spike Results (spiked by laboratory)

compound	original Sample no	spiked Sample no	concentration ADDED	concentration FOUND	concentration UNSPIKED	RELATIVE RECOVERY	LABORATORY CONTROL LIMITS	EPA CONTROL LIMITS
2,3,7,8-TCDD	M-02-14	M02-14MS	1.87	1.58	ND	84%	Not established	

ORIGINAL (Red)

* An asterisk indicates values outside control limits.

Comments: Acceptable relative recovery.

Duplicate Analysis Results

compound	Type of duplicate (Inter/Intra-Lab)	Sample No. /Lab Name	Concentration	Sample No /Lab Name	Conc.	Relative Percent Difference
2,3,7,8-TCDD	interlab	M02-13 Battelle	ND/DL 0.27 ppb	M-02-13 SPLIT/ MEAD	ND/DL 0.20	0%
2,3,7,8-TCDD	intralab	M02-13 SPLIT/ MEAD	ND/DL 0.70 ppb	M-02-13 SPLIT/ MEAD	ND/DL 0.22 ppb	0%
2,3,7,8-TCDF	interlab	M02-13/ Battelle	ND/DL 0.27 ppb	M-02-13/ Enviredyne	1.04 ppb	200%

Control limits: _____ Source of QC Limits: _____

* An asterisk indicates outliers.

Comments: Battelle attempted to analyze both Mead and Enviredyne samples at 10 grams initially. When precipitation of extracts occured, a 1g aliquot of Enviredyne (EPA) sample was analyzed. There was no successful intralab duplicate analysis done by Battelle. Enviredyne was the only lab which obtained a positive result for this sample.

(lied)

Qualitative Requirements

A. 1. Isomer Specificity Demonstrated in Documentation? (Y/N) Yes

2. Isomer Specificity Demonstrated in Documentation within 8 hours to all positive sample runs? (Y/N) Yes

Exceptions: None

B. 1. 320/322 Ion Ratio within QC Limits (.67 - .87) for all positives?

Y/N Yes Exceptions Only positives were Spike and performance audit sample.

C. 1. 320, 322, 257 All maximize together (within 3 seconds)? (Y/N) Yes

Exceptions None. Difficult to determine since retention times not printed on chromatograms. Judged by peak width versus apex separation.

2. S/N greater than 2.5 for each ion? (Y/N) Yes Exceptions: None

D. Retention time of surrogates and internal standard same as native TCDD? (Y/N) Yes Exceptions Same as (C.1.) above.

E. Confirmation Data

1. At least one confirmed per set of 24? (Y/N) Yes Exceptions

2. High resolution confirmation? (Y/N) Yes Comments All analyses were performed by Hi-Resolution GC/MS. Verbal conversation with Dr. Fred VeRoos on 11/14 indicate Resolution at mass 320 was about 30mmu or greater.

3. Partial scan confirmation? (Y/N) No.

→ Ion Ratios: QC Limits:

320/322

320/324

257/259

194/196

160, 161, 194, 196, 257, 259, 320, 322, 324

Comments Partial scan confirmation could perhaps be performed on Enviroline extract to determine what artifact caused the apparent TCDD peak in their low resolution analysis of MOZ-13. Extract may no longer be viable, though, so success is not guaranteed.

- Resolution was good enough to distinguish between 1 sulfur and 2 oxygen in MW of 320 ions.

Calibration Standards

Calibration data provided for 3 concentration levels? (Y/N) Yes
Exceptions: Multi-level calibration run before instead of after check standard!!! However since sample of interest was ND, no effect on validity of data.
Linearity verified within working range? (RRF < 10% RSD) Yes
Exceptions: None

Calibration Check data provided for all sample runs? (Y/N) Yes
Exceptions: None

Check standard RRF's within ±10% of multilevel calibrations? (Y/N) Yes
Exceptions: None

Average RRF from calibration used in all calculations? (Y/N) Yes
Exceptions: None

CALIBRATION LOG

EQUIVALENT PPB LEVEL OF TCDD	INSTRUMENT IDENTIFIER	RUN FILE IDENTIFIER	DATE/TIME OF INJECTION	RESPONSE FACTORS: 2,3,7,8-TCDD	$^{37}Cl_4$-2,3,7,8-TCDD	ISOMER STD, CHECK STD or MULTILEVEL
1.0	VG 70-70H HRMS	320525	10/25	1.01	1.35	MULTI-LEVEL
1.0		320526		1.00	1.32	
1.0		320527		0.99	1.26	
5		320528		0.89	1.24	
5		320529		0.92	1.24	
5		320530		0.94	1.24	
25		320531		0.91	1.17	
25		320601		0.95	1.17	
25		320602		1.01	1.17	
1.0		219915	10/19	0.96	1.21	Check std.

Performance Audit Results

Perf. Audit Batch ID: 3.1
Source and Preparation: Region VII EPA soil, Blended by U of Nevada for EMSL-LV
Date Prepared: 6/25/83
Analyte and Matrix: 2,3,7,8-TCDD
Interferents added: None

ORIGINAL (Red)

Reference Analysis Results (Received to date):

P.A. Batch I.D.: 3.1															
Analyte: 2,3,7,8-TCDD															
SAS (Sample Batch):	619C	619C	619C	629C	629C	630C	631C	640C	641C	642C	643C	656C	657C	691C	
Date Analyzed:	8/16	8/13-14	8/13-14	7/14	7/14	8/1-2	8/19	8/4-5	8/18	8/27-28	8/16	8/10	8/5-8	7/31-8/1	
LABORATORY:	C	V	V	W	W	V	C	W	W	C	C/T	C	V	B	
SAMPLE NO:	M-0405	M-02-15	M02-15	M03-16	M03-16	M04-13	R02-26	W-02-12	W-03-09	W-04-10	W-04-08	R01-35	R03-23	R01-37	
RESULT:	3.5	3.3	3.6	3.07	2.86	5.3	3.89	2.85	2.35	2.24	3.42	3.8	4.8	3.75	
SAMPLE NO:	M01-06	M02-16	M02-16	M03-17	—	M04-14	R02-27	W-02-13	W03-10	W04-11	W04-09	R01-36	R03-24	—	
RESULT:	3.6	3.1	3.4	3.27	—	5.4	2.86	3.01	2.64	2.19	2.73	2.9	4.1	—	
MEAN:	3.55	3.35	←	3.07	←	5.35	3.875	2.93	2.50	2.215	3.075	3.35	4.45	3.75	
Difference:	0.10	0.20	←	0.20	←	0.10	0.03	0.16	0.29	0.05	0.69	0.90	0.70	—	

ANALYTE/Perf. Audit Batch: 2,3,7,8-TCDD in 3.1
STATISTIC MEASURED: individual results
Number of values: 26
mean: 3.5
Standard deviation: 0.8

Performance Audit Sample Results:

Performance Audit Batch I.D.:	3.1												
Sample no.:	M-02-16												
compound:	2,3,7,8-TCDD												
concentration:													
mean value of audit pair (this batch):	3.47												
this lab's preceding mean (last batch):	3.75												
(2.58σ) control Limits for mean (this batch):	within	Limits											
(1.96σ) control Limits for consecutive outliers:	within	Limits											
Audit Pair difference:	—												
(RPD) for (this batch) audit pair:	—												
RPD for this lab's last batch:	—												
(2.58σ) control limits for RPD (this batch):	—												
(1.96σ) control limits for RPD consecutive:	—												

*An asterisk indicates values beyond 1.96 standard deviations from the mean.
**A double asterisk indicates values beyond 2.58 standard deviations from the mean.
NE = Not established due to insufficient data.

Comments:

ORIGINAL (red)

Calculation Check:

		calculated value		reported value
m02-16: (Performance Audit)	$\frac{(8950.35+11533.43)}{(6492.87+8253.64)} \times \frac{2.5}{10.4} \times \frac{}{0.96}$	3.47	=	3.47
m02-13: (Detection Limit)	$\frac{11.78}{2798.36} \times \frac{2.5}{1.0} \times \frac{2.5}{0.96}$	0.274 ppb D.L.	≠	0.83 ppb D.L.

- The discrepancy in the calculated versus reported detection limits arises out of the interpretation of the section of the dioxin protocol which addresses calculation of detection limits. Two chemists, Paul Taylor, pHD., President of Cal. Analytical, and Angelo Carasea, Region VII EPA contributing author of the protocol, agreed with this reviewer in the following interpretation: When interfering peaks greater than 2.5 times the noise level are present in the 2,3,7,8-TCDD retention window for both masses 320 and 322, and if one interference is significantly larger than the other, then the detection limit can be stated (conservatively) as 2.5 times the amount calculated by the lower level interfering mass area and the corresponding C_{13}-TCDD mass area. (This is different than the reported detection limit, which was calculated using the sum of the two masses 320 and 322, and which yielded a higher result due to a relatively higher interference at mass 322 versus mass 320.) Mass chromatograms of this sample have been attached to illustrate the observed phenomena.

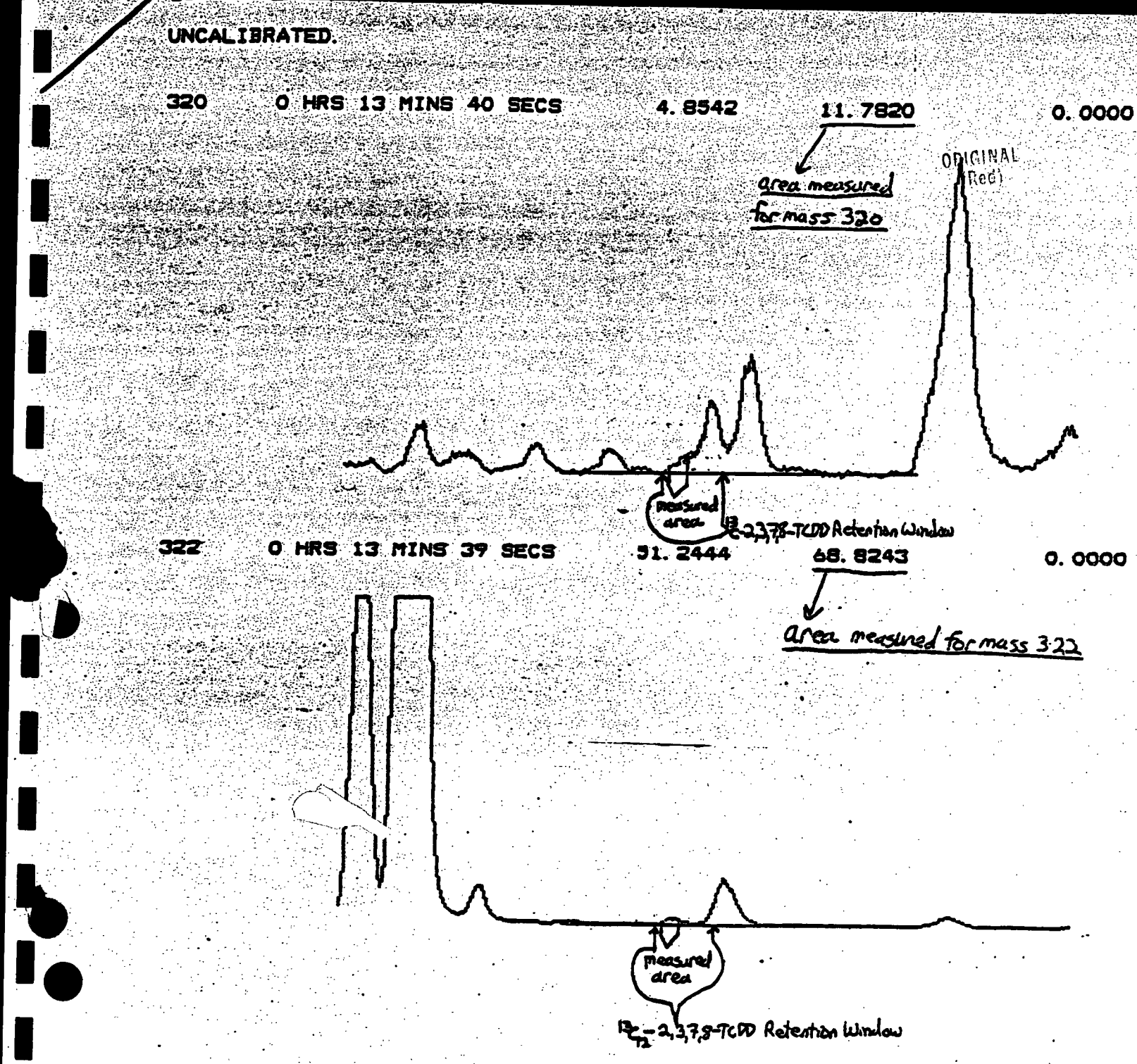


FIGURE 5-A. SELECTED ION CURRENT TRACE FOR m/z 320 AND m/z 322 FOR SAMPLE M-02-13

PROJECT NAME: FMC Agricultural
TDD NO: F3-8306-20

EPA SITE NO.: M-02
REGION: VII

ORIGINAL (Red)

QUALITY ASSURANCE REVIEW OF DIOXIN ANALYSIS LAB DATA PACKAGE

Case No./SAS No.: Not EPA funded
Contract No.: " " "
Contract Laboratory: Mead Comp/chem
Analytical Protocol: Region VII soil dioxin
Reviewer: R. Slofada
Review Date: 11/15/83

Applicable Sample No's.: Mead Comp/chem sample 13228, which was a split sample of M-02-13.

The dioxin analytical data for this case has been reviewed. The quality assurance evaluation is summarized in the following table:

Reviewer's Evaluation*	Fraction				
	2,3,7,8-TCDD	Other TCDD's	Other chlorinated dibenzodioxins	2,3,7,8-TC dibenzofuran	Other Cl'd dibenzofurans
Acceptable		Not analyzed →			
Acceptable with exception(s)	✓ 1				
Questionable					
Unacceptable					

* Definitions of the evaluation score categories are listed on next page.

This evaluation was based upon an analysis of the review items indicated below:

- ☑ DATA COMPLETENESS [1]
- ○ BLANK ANALYSIS RESULTS
- ☑ SURROGATE SPIKE RESULTS
- ○ MATRIX SPIKE RESULTS
- ☑ DUPLICATE ANALYSIS RESULTS
- ☑ QUALITATIVE REQUIREMENTS
- ☑ CALIBRATION STANDARDS
- ○ PERFORMANCE AUDIT RESULTS
- ☑ CALCULATION CHECKS

Data review forms are attached for each of the review items indicated above.

Comments: [1] Data package was not as complete as the Region VII EPA protocol requires. However, essential chromatograms were provided with a brief narrative. Although not all deliverables present, if lab was following Region VII protocol during analysis one can conclude that data rules out the presence of TCDD in the sample aliquot at the reported detection limit of 0.20 ppb. Many interferences were noted in this analysis. Additional cleanup was necessary to remove background interferences. One interfering peak, but which had a different retention time than 2,3,7,8-TCDD, had a 320/322 Ratio of 0.80, but 257 peak was not seen.

DATA EVALUATION SCORE CATEGORIES

ORIGINAL
(Red)

ACCEPTABLE: Data is within established control limits, or the data which is outside established control limits does not affect the validity of the analytical results.

ACCEPTABLE WITH EXCEPTION(S): Data is not completely within established control limits. The deficiences are identified and specific data is still valid, given certain qualifications which are listed below.

QUESTIONABLE: Data is not within established control limits. The deficiences bring the validity of the entire data set into question. However, the data validity is neither proved nor disproved by the available information.

UNACCEPTABLE: Data is not within established control limits. The deficiences imply the results are not meaningful.

TCDD DATA COMPLETENESS CHECKLIST

SAMPLE NO. Field SPLit of sample	13328 #02-13	13328 #02-13								
LAB I.D. NO.	THDD 5104C03	TRD0514 A03								ORIGINAL (Red)
MATRIX	soil →									
RUN DATE/TIME	10/6 1:39	10/7 13:30								
INSTRUMENT I.D. NO.	OWA #3	OWA #3								
TABULATED RESULTS	✓	✓								
DETECTION LIMITS	✓	✓								
SURROGATE ACCURACY	ms	ms								
ION AREAS	✓	✓								
ION RATIOS	MS	MS — But can calculate.								
MID CHROMATOGRAMS	✓	✓								
PREVIOUS RUN AREAS	NA →									
PREVIOUS RUN CHROS.	NA →									
REANALYSIS LOG	✓	✓								
3 PT. CALIB. R.F./AMTS.	ms	ms								
3 PT. CALIB. MID CHROS.	ms	ms								
DAILY CALIB. RF/AMTS.	ms	ms								
DAILY CALIB. MID CHROS.	10/5 20:20 ✓	10/7 9:04 ✓								
ISOMER SEPARATION CHROS.	✓	✓								
STANDARD SOURCE	ms	ms								
EXTRACTION WT.	ms	ms								
CLEANUP METHOD	ms	ms								
CALCULATION VOLUMES	✓	✓								
PARTIAL SCAN SPECTRA										
HIGH RESOLUTION DATA										
LAB SPIKE RECOVERY			ms							
LAB DUPLICATE	✓	✓								
LAB BLANK			ms							
PERFORMANCE AUDIT SPL.										
INTER-LAB. DUPLICATE										
SAMPLE BLANK										
DECON. RINSATE										

ms = missing data normally required for EPA data package for dioxin.

Blank Analysis Results

ORIGINAL
(Red)

The contaminants found in the blanks are listed below:

FRACTION	TYPE OF BLANK	SAMPLE NO.	SOURCE OF water/soil	CONTAMINANTS (concentration/DL)

COMMENTS: No blank results provided, but not important since sample results were nondetected.

SURROGATE SPIKE RECOVERIES (RELATIVE TO INT. STD.)

* Asterisked values are outside of QC limits

ORIGINAL (Red)

Surrogate compound name:		37Cl4-2,3,7,8-TCDD			
Analytical Fraction:					
QC LIMITS FOR SOILS	Laboratory C.L.:				
	EPA Action:	60-140			
	source:	Ref. 1	Ref.	Ref.	Ref.
QC LIMITS FOR WATER	Laboratory C.L.:				
	EPA ACTION:				
	source:	Ref.	Ref.	Ref.	Ref.
Matrix	Sample no.				
Soil	m-02-13 #1	*1			
Soil	m-02-13 #2	*2			

Surrogate compound name:						
Analytical Fraction:						
QC LIMITS FOR SOILS	Laboratory C.L.:					
	EPA Action:					
	source:	Ref.	Ref.	Ref.	Ref.	Ref.
QC LIMITS FOR WATER	Laboratory C.L.:					
	EPA ACTION:					
	source:	Ref.	Ref.	Ref.	Ref.	Ref.
Matrix	Sample no.					

Source of QC Limits: Ref. 1: Region VII soil protocol, September '83 revision.
Ref. 2:

COMMENTS: *1 = area Ratio $\frac{328}{332+332} = \frac{181440}{179584+218624} = .45$ assuming 25 ng I.S., 10 ng Surr, RF calculates to 1.14, which is similar to what other labs have reported. Thus, recovery seems reasonable. *2 = area ratio = 317424/(291456+368064) = .48 assuming 25 ng I.S., 10 ng Surr, RF calculates to 1.20, which is similar to what other labs have reported. Assuming that region VII protocol amts. were added, it appears surrogate accuracy or relative recovery is reasonable, probably 100% plus or minus 40%.

Matrix Spike Results (spiked by laboratory)

compound	original Sample no	spiked Sample no	concentration ADDED	concentration FOUND	concentration UNSPIKED	RELATIVE RECOVERY	LABORATORY CONTROL LIMITS	EPA CONTROL LIMITS

* An asterisk indicates values outside control limits.

Comments: Matrix spike results not provided in data package.

Duplicate Analysis Results

compound	Type of duplicate (Inter/Intra-Lab)	Sample No. / Lab Name	concentration	Sample No / Lab Name	conc.	Relative Percent Difference
$^{37}Cl_4$-2,3,7,8-TCDD	intralab	split of mo2-13 Run #1	RF=1.14	split of mo2-13 Run #2	RF=1.20	5.1%
2,3,7,8-TCDD	intralab	split of mo2-13 Run #1	ND/DL=0.7ppb	split of mo2-13 Run #2	ND/DL=0.2ppb	—
2,3,7,8-TCDD	interlab	mo2-13 Battelle	ND/DL=0.27ppb	mo2-13 Enviredyne	1.04ppb	200%

Control limits: _____ Source of QC Limits: _____

* An asterisk indicates outliers.

Comments: Enviredyne was the only lab to obtain a positive result for this sample. Battelle and Enviredyne analyzed mo2-13. Mead analyzed mo2-13 field split. The sample was homogenized in the field (using blender) before splitting. Consequently, MEAD sample should be very similar in composition to mo2-13.

ORIGINAL (Red)

Qualitative Requirements

A.1. Isomer Specificity Demonstrated in Documentation? (Y/N) No

2. Isomer Specificity Demonstrated in Documentation within 8 hours to all positive sample runs? (Y/N) No

Exceptions: No data provided.

B.1. 320/322 Ion Ratio within QC Limits (.67 - .87) for all positives?

Y/N NA Exceptions No Positives in this set at this laboratory

C.1. 320, 322, 257 All maximize together (within 3 seconds)? (Y/N) NA

Exceptions No positives in this set, but surrogate and internal standard masses within 3 seconds of each other.

2. S/N greater than 2.5 for each ion? (Y/N) Yes Exceptions: None

Could not see height of surrogate in Run #1 Chromatogram, but can tell from labelled areas that it was > 2.5 noise since no peaks nearby.

D. Retention time of surrogates and internal standard same as native TCDD?

(Y/N) Yes Exceptions: For sample run #1, I.S. was within 1 second of daily standard. For Run #2, internal standard was within 2 seconds of standard run.

E. Confirmation Data

1. At least one confirmed per set of 24? (Y/N) NO Exceptions

2. High resolution confirmation? (Y/N) Yes Comments High resolution ND run contradicts TCDD found in EPA's initial analysis done by Enviodyne. Earlier result may be artifactual, or difference may be due to inhomogeneity.

3. Partial scan confirmation? (Y/N) No

→ Ion Ratios: QC Limits:

320/322 _____

320/324 _____

257/259 _____

194/196 _____

160, 161, 194, 196, 257, 259, 320, 322, 324 _____

Comments Data sufficient to rule out the 2,3,7,8-Isomer, provided protocol was followed. Detection limit is roughly verified to one significant figure.

Calibration Standards

Calibration data provided for 3 concentration levels? (Y/N) No ORIGINAL (Red)
Exceptions: No data provided

Linearity verified within working range? (RRF < 10% RSD) Cannot determine - no data
Exceptions:

Calibration Check data provided for all sample runs? (Y/N) Incomplete --
Exceptions: Only RIC chromatograms of standards provided.

Check standard RRF's within ±10% of multilevel calibrations? (Y/N) Cannot determine
Exceptions:

Average RRF from calibration used in all calculations? (Y/N) Cannot determine
Exceptions:

CALIBRATION LOG

EQUIVALENT LEVEL OF TCDD	INSTRUMENT IDENTIFIER	RUN FILE IDENTIFIER	DATE/TIME OF INJECTION	RESPONSE FACTORS: 2378-TCDD	$^{37}Cl_4$-2378-TCDD	ISOMER STD, CHECK STD or MULTILEVEL
If done by Protocol probably ~1ppb.)	OWA #3	H283100S803	10/5 22:06	?	?	Check Std.
" " "	OWA #3	HS83100TA03	10/7 9:14	?	?	check std.

- Calculation Check: Rough Estimate to Verify Detection Limit

Run #2: → $\frac{(672+1120)}{(33248+41952)} \frac{25}{10} \frac{2.5}{0.60} = 0.25$ ppb detection limit calculated estimate
≈ 0.20 detection limit reported.

(Correct to one significant figure since RRF should be greater than 0.50.)

estimated sample weight; estimated RRF

Run #1: → Cannot verify detection limit because Chromatogram not to scale for measuring noise at mass 320, the lower noise ion.

ORIGINAL
(Red)

Appendix E

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 3
Curtis Bldg., 6th & Walnut Sts.
Philadelphia, Pennsylvania 19106

CHAIN OF CUSTODY RECORD

PROJ. NO.	PROJECT NAME
83-5301-20	[illegible] -02

SAMPLERS: *(Signature)* D. J. E. [illegible]
[illegible] for Richard M. [illegible]

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	[illegible]						REMARKS
[illegible]-02-01	6/21/83	1040		x	Facility - North Side	1	1						Tag #3-13550
[illegible]-02-02	6/21/83	1111		x	Facility - North Side	1	1						Tag #3-13718
[illegible]-02-03	6/21/83	1138		x	Facility - North Side	1	1						Tag #3-14593
[illegible]	6/21/83	1206		x	Facility - South Side	1	1						Tag #3-14579
[illegible]-05	6/21/83	1307		x	Facility - North Side	1	1						Tag #3-12051
[illegible]	6/21/83	13[illegible]		x	Facility - North Side	1	1						Tag #3-~~[illegible]~~ 12052 (DJS)
[illegible]-07	6/21/83	1326 ~~1324~~ (TJS)		x	Facility - North Side	1	1						Tag #3-12087
[illegible]-2-08	6/21/83	1340		x	Facility - North Side	1	1						Tag #3-12094
[illegible]-09	6/21/83	13[illegible]		x	Facility - South Side	1	1						Tag #3-12096
[illegible]	6/21/83	1519		x	Facility - South Side	1	1						Tag #3-13098
[illegible]-12	6/21/83	1638		x	Facility - South Side	1	1						Tag #3-24051
[illegible]-13	6/21/83	1655		x	Facility - South Side	1	1						Tag #3-24057
[illegible]	[illegible]	1712		x	Facility - South Side	1	1						Tag #3-24060
[illegible]	6/21/83	1717		x	Facility - South Side	1	1						Tag #3-24061

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Mike Nalipinski	6/24/83 5:00pm	Russell J. [illegible] *	Russell J. [illegible]	6/29/83 5:00pm	
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*

Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Remarks

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

* Received sample from Mike Nalipinski, stored in custody at office until relinquished.

3-10878

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 3
Curtis Bldg., 6th & Walnut Sts.
Philadelphia, Pennsylvania 19106

CHAIN OF CUSTODY RECORD

PROJ. NO.	PROJECT NAME
F3-8306-28	M-02

SAMPLERS: *(Signature)* Ronald J. Gilbert

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	40 ml vial	REMARKS
M-02-15	6/29/83	4:30p		X	Facility = shipping area	1		Log # 3-24901
M-02-16	6/29/83	4:40p		X	Facility = shipping area	1		Log # 3-24902

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Ronald J. Gilbert	6/29/83 5:00p				
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

3-11571

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 3
Curtis Bldg., 6th & Walnut Sts.
Philadelphia, Pennsylvania, 19106

CHAIN OF CUSTODY RECORD

PROJ. NO.	PROJECT NAME
F3-83.06-LB	M-01

SAMPLERS: (Signature) Jeffrey J. Carr

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	1/4 OZ JAR					REMARKS
M01-1	6/21/83	1055		X	Hexachlorophene	1	1					TAG # 3-39063

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Bill Westervelt	6/23/83 0650	[illegible]	Terry Shannon *	6/24 5:04p	[illegible]
[illegible]	6/24/83 5:00p				

Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

* R. Slobodin received samples from T. Shannon at 6/24 5:00pm; samples stored in custody at office until Relinquished

3-10887

FMC Agricultural Chemical Group
Baltimore MD

SECRECY AGREEMENT

ORIGINAL
(Red)

Many of the operations in our plant involve highly confidential information, which we are treating as trade secrets. Before allowing you into our plant, therefore, we must have your agreement to treat anything you observe as confidential. In no event will you reveal to anybody anything that you observe in our plant, whether new to you or not. Moreover, you agree to hold as confidential and not to disclose or use for any purpose any information that you receive or learn in our plant, which was not previously known to you, or which is not public knowledge, or which you do not receive from some other source at a future time.

I have read, understand, and agree to abide by the above listed agreement.

Signed Donald Senovich Date 6/20/83

Print Name DONALD SENOVICH Witnessed By Doug W. Palmer

Representing NUS CORP.

Form 60 1/7/81

FEDERAL EXPRESS

904211055 (pink)

YOUR FEDERAL EXPRESS ACCOUNT NUMBER	1059-6338-6
DATE	6/29/83

FROM (Your Name)	TO (Recipient's Name)
Russell J. Sloboda	Judy Stone
COMPANY: NUS Corporation	COMPANY: Envirodyne
STREET ADDRESS: 942 Old Eagle School Rd. Suite 916	STREET ADDRESS (P.O. BOX NUMBERS ARE NOT DELIVERABLE): 12161 Lakeland Rd.
CITY: Wayne STATE: Pennsylvania	CITY: St. Louis STATE: Missouri
ZIP: 19087	ZIP: 63141

AIRBILL NO. 76884433

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PACKAGES	WEIGHT	DECLARED VALUE
1	40	
1	40	
TOTAL 2	TOTAL 80	TOTAL

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Federal Express Corporation Employee No. 32174

DATE/TIME For Federal Express Use: 6-29-83

FEDERAL EXPRESS USE: FREIGHT CHARGES; DECLARED VALUE CHARGE; ADV. ORIGIN; ADV. DEST.; ADVANCE ORIGIN; ADVANCE DESTINATION; OTHER; TOTAL CHARGES

PART #2041730764 REVISION DATE 10/82 PRINTED U.S.A.

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(TYPE OR PRINT)

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ADDITIONAL DESCRIPTION REQUIREMENTS FOR RADIOACTIVE MATERIALS (SEE BACK)	RADIONUCLIDE	FORM	ACTIVITY	CATEGORY OF LABELS	TRANS. INDEX	PACKAGE IDENTIFICATION

THIS SHIPMENT IS WITHIN THE LIMITATIONS PRESCRIBED FOR PASSENGER AIRCRAFT / CARGO AIRCRAFT ONLY (DELETE-NONAPPLICABLE)

IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS OR TREATMENT.

I HEREBY CERTIFY THAT THE CONTENTS OF THIS CONSIGNMENT ARE FULLY AND ACCURATELY DESCRIBED ABOVE BY PROPER SHIPPING NAME AND ARE CLASSIFIED, PACKED, MARKED, AND LABELED, AND IN PROPER CONDITION FOR CARRIAGE BY AIR ACCORDING TO APPLICABLE NATIONAL GOVERNMENTAL REGULATIONS.

NAME AND TITLE OF PERSON SIGNING CERTIFICATION	EMERGENCY TELEPHONE NO.	SIGNATURE OF SHIPPER
Russell J. Sloboda, Chemist, NUS Corp.	215 687 9510	

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number: 619C

SPECIAL ANALYTICAL SERVICE
PACKING LIST

Sampling Office:	Sampling Date(s):	Ship To:	For Lab Use Only
III	6/21/83	Envirodyne 12161 Lakeland Rd. St. Louis, Mo. 63141	Date Samples Rec'd:
Sampling Contact: (name)	Date Shipped:		
T. Shapiro	6/29/83		Received By:
215-687-9510 (phone)	Site Name/Code: m-02	Attn: Judy Stone	

	Sample Numbers	Sample Description i.e., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1.	m-02-01	2,3,7,8-TCDD Analysis / Solid / Low	
2.	m-02-02	2,3,7,8-TCDD Analysis / Solid / Low	
3.	m-02-03	2,3,7,8-TCDD Analysis / Solid / Low	
4.	m-02-04	2,3,7,8-TCDD Analysis / Solid / Low	
5.	m-02-05	2,3,7,8-TCDD Analysis / Solid / Low	
6.	m-02-06	2,3,7,8-TCDD Analysis / Solid / Low	
7.	m-02-07	2,3,7,8-TCDD Analysis / Solid / Low	
8.	m-02-08	2,3,7,8-TCDD Analysis / ~~Solid~~ Aqueous (TAS) / Low	
9.	m-02-09	2,3,7,8-TCDD Analysis / Solid / Low	
10.	m-02-10	2,3,7,8-TCDD Analysis / Solid / Low	
11.	m-02-11	2,3,7,8-TCDD Analysis / Solid / Low	
12.	m-02-13	2,3,7,8-TCDD Analysis / Solid / Low	
13.	m-02-14	2,3,7,8-TCDD Analysis / Solid / Low	
14.	m-02-14'	2,3,7,8-TCDD Analysis / Solid / Low / "To Be Spiked By Laboratory"	
15.			
16.	m-01-01	2,3,7,8-TCDD Analysis / ~~Solid~~ Aqueous (TAS) / High	
17.		Note: Sample is hexachlorophene powder.	
18.	m-02-15	2,3,7,8-TCDD Analysis / Solid / Low	
19.	m-02-16	2,3,7,8-TCDD Analysis / Solid / Low	
20.			

For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

On June 21, 1983, C.A. Shaheen gave permission for Terrance A. Shannon, of NUS Corp. to remove property from their facility in the following containers: 15- one pint jars for 2,3,7,8- TCDD analysis, and 24- eight ounce jars.

C.A. Shaheen — 6/21/83

Facility Representative — Date

Terrance A. Shannon — 6/21/83

NUS Representative — Date

Appendix F

TABLE 1

DATA SUMMARY

Lab: Envirodyne Engineers, Inc.

Date: July 22, 1983

Case: 619C

GC Column[a]: SP-2330, 60 meters
SP-2340, 60 meters

Sample Number	Extraction	Cleanup	Wet Weight (grams)	TCDD (ppb)	D.L. (ug/kg)	320/322	Surrogate Percent Recovery	257	320	322	328[b]	332	334	FRN[c] Number
M-02-01	J	A,D	10.5	-	0.63	-	111	-	-	-	414	346	474	23601
M-02-02	J	A	10.3	-	0.22	-	93	-	-	-	999	1106	1349	23525
M-02-03	J	A	10.1	-	0.20	-	83	-	-	-	678	843	1032	23526
M-02-04	J	A,D	10.3	-	0.21	-	102	-	-	-	741	742	922	23624
M-02-04D	J	A,D	10.1	-	0.14	-	101	-	-	-	629	662	767	23625
M-02-05	J	A	10.1	-	0.06	-	85	-	-	-	1306	1550	1955	23529
M-02-06	J	A,D	10.8	-	0.15	-	95	-	-	-	1512	1564	1919	23602
M-02-07	J	A,D	10.6	-	0.19	-	91	-	-	-	528	591	738	23623
M-02-09	J	A	11.1	-	0.47	-	92	-	-	-	810	902	1110	23533
M-02-10	J	A,D	10.4	-	0.14	-	98	-	-	-	1698	1698	2116	23604
M-02-12	J	A,D	10.7	-	0.69	-	93	-	-	-	193	203	272	23620
M-02-13	J	A,D	10.4	-	Interferences, is being re-extracted and reanalyzed; See FRNs 23536 and 23621.									
M-02-14	J	A,D	10.8	-	0.46	-	104	-	-	-	111	99	145	23622
M-02-14N	J	A	10.1	1.2	-	0.73	93	142	352	481	895	999	1208	23539[d]
M-02-14N[a]	J	A,D	10.1	1.3	-	0.82	104	462	1103	1340	2618	4263	5330	6912
M-02-15	J	A	10.1	3.3	-	0.82	94	336	723	880	639	706	845	23541[d]
M-02-15[a]	J	A	10.1	3.6	-	0.74	113	731	2376	3226	2042	3290	4340	6911[d]
M-02-16	J	A	10.1	3.1	-	0.79	95	682	1529	1933	1452	1556	1969	23542
M-02-16[a]	J	A	10.1	3.4	-	0.78	113	839	2881	3677	2531	4201	5271	6913
MB (solls)	J	A	-	-	0.24[e]	-	83	-	-	-	646	797	989	23545
M-01-01 (powder)	J	A,B	10.1	-	0.20	-	99	-	-	-	308	329	387	23547
MB (powder)	J	A,B	-	-	0.84[e]	-	93	-	-	-	85	95	114	23548
M-02-08 (rinsate)	-	A	65[f]	-	0.039[g]	-	95	-	-	-	1297	1436	1697	23532

ORIGINAL (Red)

TABLE 1

DATA SUMMARY

Lab: Envirodyne Engineers, Inc.

Date: July 22, 1983

Case: 619C

GC Column[a]: SP-2330, 60 meters
SP-2340, 60 meters

Sample Number	Extraction	Cleanup	Wet Weight (grams)	TCDD (ppb)	D.L. (ug/kg)	320/322	Surrogate Percent Recovery	257	320	322	328[b]	332	334	FRN[c] Number

Notes: MB = Method Blank
J = Jar Extraction
A,B,C = Cleanup Option from Method
FRN = File Refence Number
D = Duplicate
D.L. = Detection Limit
N = Native 2,3,7,8-TCDD Spike

[a]Samples were all initially analyzed on SP-2340 column. Positive values were confirmed on the SP-2330 column.
[b]Corrected for contribution by native TCDD (subtracted 0.009 of m/e 322).
[c]Samples with FRNs 23xxx analyzed on HP-5985 (SP-2340 column). Samples with FRNs 69xx were analyzed on HP-5993 (SP-2330 column).
[d]Samples run just prior to these positives:

Positive Sample	FRN	Previous Sample	FRN
M-02-14N	23539	M-02-14	23538
M-02-15	23541	Mixed Isomer standard	23540
M-02-15 confirmation	6911	Another project	6910

[e]Assumes 10 g sample.
[f]In milliliters
[g]In ug/l.

ORIGINAL
(Red)

TABLE 1 - DATA SUMMARY

Lab: Envirodyne Engineers, Inc.

Date: 9/20/83

Case: 619C Rerun

GC Column: SP-2330, 60 meter

Sample Number	Extraction	Cleanup	Wet Weight (grams)	TCDD (ug/kg)	D.L. (ug/kg)	Analytical Date	Analytical Time	320/322	332/334	Surrogate Percent Accuracy	257	320	322	328[a]	332	334	FRN Numbers
619C-M02-13	J	A,D	10.4	1.0	-	8/26	0040	0.67	0.86	80	58	87	130	250	280	328	7384

Notes: MB = Method Blank
J = Jar Extraction
A,B,C,D = Cleanup Option from Method
FRN = File Reference Number
D = Duplicate
D.L. = Detection Limit
N = Native 2,3,7,8-TCDD Spike

[a]Corrected for contribution by native TCDD (subtract 0.009 of m/e 322).

ORIGINAL (Red)

Lab: Battelle

Case:

Shipment:

Date: 10/19/83

GC Column: CP SIL-88

Average Native RRF: 0.95

Average Surrogate RRF: 1.24

Sample Number	Extraction Method	Cleanup Option	Gm. Wt.	PPB TCDD	D.L.	Analytical Date	Time	Native m/z 320 / m/z 322 Area Ratio	C-13 m/z 332 / m/z 334 Area Ratio	Surrogate (1) Percent Accuracy	m/z 320 area	m/z 322 area	m/z 257 area	m/z 328 area	m/z 332 area	m/z 334 area	Comments
13228	J	B	10.8			10/19				UNABLE TO ANALYZE DUE TO PRECIPITATE							
M-02-13	J	B	10.8			10/19				"							
M-02-14	J	B	10.9	ND	.02	10/19	--	.95	.80	120	31.90	33.53	--	7711.42	7091.71	8917.86	
M-02-16	J	B	10.4	3.47	--	10/19	--	.78	.78	121	8950.35	11533.43	--	7235.18	6499.87	8283.64	
M-02-14N	J	B	10.7	1.58	--	10/19	--	.78	.80	119	4819.68	6141.26	--	8358.66	7767.68	9703.96	
MB	J	B	10.0	0.01	--	10/19	--	.77	.81	119	27.01	35.16	--	7026.23	6582.39	8158.30	
M-02-13	J	B	1.0	ND	.83	10/19	--	.17	.76	120	11.78	68.82	--	3049.33	2798.36	3544.60	

(1) Corrected for contribution by native TCDD (Subtract 0.009 of m/z 322).
(2) Based on 10 gram sample.

MB = Method Blank
P = Partial Scan
N = Native TCDD Spike
D = Duplicate (Intralab)
FB = Field Blank

H = High Resolution
ND = Not Detected
DL = Detection Limit
J = Jar Extraction

A,B,C = Cleanup Option (or any combination)

ORIGINAL (Red)

ORIGINAL
(Red)

EXHIBIT II -COMPOUND LIST

SAMPLE IDENTIFIER: #13
COMPUCHEM SAMPLE NUMBER: 13228 2nd analysis

COMPOUND	RESULT (ppb)	DETECTION LIMIT (ppb)
2378-TCDD	Not Detected	0.20

ORIGINAL
(Red)

EXHIBIT II -COMPOUND LIST

SAMPLE IDENTIFIER: #13
COMPUCHEM SAMPLE NUMBER: 13228 1st analysis

COMPOUND	RESULT (ppb)	DETECTION LIMIT (ppb)
2378-TCDD	Not Detected	0.70

Appendix E

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 3
Curtis Bldg., 6th & Walnut Sts.
Philadelphia, Pennsylvania 19106

CHAIN OF CUSTODY RECORD

PROJ. NO.: F3-8306-20

PROJECT NAME: M-02

SAMPLERS: *(Signature)* D. J. F. H[illegible]k; Mike Nalipinski; Richard M. Connors

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	1 lit jar						REMARKS
M-02-01	6/21/83	1040		X	Facility - North Side	1	1						Tag # 3-15550
M-02-02	6/21/83	1111		X	Facility - North Side	1	1						Tag # 3-15718
M-02-03	6/21/83	1138		X	Facility - North Side	1	1						Tag # 3-14593
M-02-04	6/21/83	1206		X	Facility - South Side	1	1						Tag # 3-14599
M-02-05	6/21/83	1337		X	Facility - North Side	1	1						Tag # 3-12054
M-02-06	6/21/83	1305		X	Facility - North Side	1	1						Tag # 3- ~~12091~~ 12088 (TAS)
M-02-07	6/21/83	1326 ~~1324~~ (TNS)		X	Facility - North Side	1	1						Tag # 3-12091
M-02-08	6/21/83	1340		X	Facility - North Side	1	1						Tag # 3-12094
M-02-09	6/21/83	1322		X	Facility South Side	1	1						Tag # 3-12095
M-02-11	6/21/83	1549		X	Facility South Side	1	1						Tag # 3-12098
M-02-12	6/21/83	1630		X	Facility South Side	1	1						Tag # 3-24054
M-02-13	6/21/83	1648		X	Facility South Side	1	1						Tag # 3-24057
M-02-14	6/21/83	1712		X	Facility South Side	1	1						Tag # 3-24060
M-02-14	6/21/83	1717		X	Facility South Side	1	1						Tag # 3-24061

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Mike Nalipinski	6/24/83 5:00pm	Russell J. Sl[illegible] *	Russell J. Sl[illegible]	6/29/83 5:00pm	
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*

Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Remarks

ORIGINAL (Red)

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

* Received sample from Mike Nalipinski, stored in custody at office until relinquished.

3-10878

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 3
Curtis Bldg., 6th & Walnut Sts.
Philadelphia, Pennsylvania 19106

CHAIN OF CUSTODY RECORD

PROJ. NO.	PROJECT NAME
F3-8306-20	M-02

SAMPLERS: (Signature) Ronald J. Gilbert

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	40 ml vial	REMARKS
M-02-15	6/29/83	4:30 pm		X	Facility - shipping area	1		tag # 3-24901
M-02-16	6/29/83	4:40 pm		X	Facility - shipping area	1		tag # 3-24902

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Ronald J. Gilbert	6/29/83 5:09 pm				
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	

ORIGINAL (Red)

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

3-11571

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 3
Curtis Bldg., 6th & Walnut Sts.
Philadelphia, Pennsylvania 19106

CHAIN OF CUSTODY RECORD

PROJ. NO.: F3-8306-1B
PROJECT NAME: M-01
SAMPLERS: (Signature) [signature]

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	16 oz JAR						REMARKS
													TAG #
M01-1	6/21/83	1055		X	Hexachlorophene	1	1						3-24064
		DH											

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Bill Wentworth	6/23/83 0650	[signature]	Terry Shannon *	6/24 5:00pm	[signature]
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
[signature]	6/24/83 5:00pm				
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

ORIGINAL (Red)

* R. Slobodian received samples from T. Shannon at 6/24 5:00pm, samples stored in custody at Eflo until Relinquished

3-10887

OA Dup Transfer

FMC Agricultural Chemical Group
Baltimore MD

SECRECY AGREEMENT

ORIGINAL
(Red)

Many of the operations in our plant involve highly confidential information, which we are treating as trade secrets. Before allowing you into our plant, therefore, we must have your agreement to treat anything you observe as confidential. In no event will you reveal to anybody anything that you observe in our plant, whether new to you or not. Moreover, you agree to hold as confidential and not to disclose or use for any purpose any information that you receive or learn in our plant, which was not previously known to you, or which is not public knowledge, or which you do not receive from some other source at a future time.

I have read, understand, and agree to abide by the above listed agreement.

Signed Donald Senovich Date 6/20/83

Print Name DONALD SENOVICH Witnessed By Dana W. Palmer

Representing NUS CORP.

Form 60 1/7/81

FEDERAL EXPRESS

SHIPPERS COPY

SHIPPER: ALWAYS REFER TO BOTH ORIGINAL AIRBILL NUMBER AND THIS CROSS REFERENCE NUMBER WHEN MAKING INQUIRIES.

904211055 (pink)

AIRBILL NUMBER 768844333

SEE BACK OF FORM SET FOR COMPLETE PREPARATION INSTRUCTIONS.

YOUR FEDERAL EXPRESS ACCOUNT NUMBER: 1059-6338-6

DATE: 6/29/83

FROM (Your Name): Russell J. Slaboda

COMPANY: NUS Corporation

DEPARTMENT/FLOOR NO.:

STREET ADDRESS: 992 Old Eagle School Rd. Suite 916

CITY: Wayne STATE: Pennsylvania

ZIP (ACCURATE ZIP CODE REQUIRED FOR CORRECT INVOICING): 19087

AIRBILL NO.: 768844333

TO (Recipient's Name): Judy Stone

If Hold For Pick-Up or Saturday Delivery, Recipient's Phone Number: ORIGINAL

COMPANY: Envirodyne

DEPARTMENT/FLOOR NO.: (Rec)

STREET ADDRESS (P.O. BOX NUMBERS ARE NOT DELIVERABLE): 12161 Lakeland Rd.

CITY: St. Louis STATE: Missouri

ZIP (ACCURATE ZIP CODE REQUIRED FOR OVERNIGHT DELIVERY): 63141

IN TENDERING THIS SHIPMENT, SHIPPER AGREES THAT F.E.C. SHALL NOT BE LIABLE FOR SPECIAL, INCIDENTAL OR CONSEQUENTIAL DAMAGES ARISING FROM CARRIAGE HEREOF. F.E.C. DISCLAIMS ALL WARRANTIES, EXPRESS OR IMPLIED, WITH RESPECT TO THIS SHIPMENT. THIS IS A NON-NEGOTIABLE AIRBILL SUBJECT TO CONDITIONS OF CONTRACT SET FORTH ON REVERSE OF SHIPPER'S COPY. UNLESS YOU DECLARE A HIGHER VALUE, THE LIABILITY OF FEDERAL EXPRESS CORPORATION IS LIMITED TO $100.00. FEDERAL EXPRESS DOES NOT CARRY CARGO LIABILITY INSURANCE.

YOUR NOTES/REFERENCE NUMBERS (FIRST 12 CHARACTERS WILL ALSO APPEAR ON INVOICE): 3

PAYMENT: ☐ Bill Shipper ☐ Bill Recipient's F.E.C. Acct. ☑ Bill 3rd Party F.E.C. Acct. ☐ Bill Credit Card ☐ Cash In Advance Account Number/Credit Card Number

FEDERAL EXPRESS USE

FREIGHT CHARGES

DECLARED VALUE CHARGE

AGT/PRO

ADVANCE ORIGIN

AGT/PRO

ADVANCE DESTINATION

OTHER

TOTAL CHARGES

PART #2041730764
REVISION DATE 10/82$
PRINTED U.S.A.

SHIPPER'S COPY

SERVICES (CHECK ONLY ONE BOX)	DELIVERY AND SPECIAL HANDLING (CHECK SERVICES REQUIRED)	PACKAGES	WEIGHT	DECLARED VALUE	O/S
PRIORITY 1 (OVERNIGHT PACKAGES) (Up to 70 LBS.)	1 ☐ HOLD FOR PICK-UP AT FOLLOWING FEDERAL EXPRESS LOCATION SHOWN IN SERVICE GUIDE. RECIPIENT'S PHONE NUMBER IS REQUIRED.	1	40		
	2 ☐ DELIVER				
	3 ☑ SATURDAY SERVICE REQUIRED See Reverse (Extra charge applies for delivery.)				
	4 ☐ RESTRICTED ARTICLES SERVICE (P-1 and Standard Air Packages only, extra charge)				
STANDARD AIR (2nd BUSINESS DAY FOLLOWING PICK-UP (UP TO 150 LBS.)	5 ☐ dSS (Signature Security Service required, extra charge applies)				
	6 ☐ DRY ICE _____ LBS.				
	7 ☐ OTHER SPECIAL SERVICE				
	8 ☐				
	9 ☐	TOTAL	TOTAL	TOTAL	
		1	40		

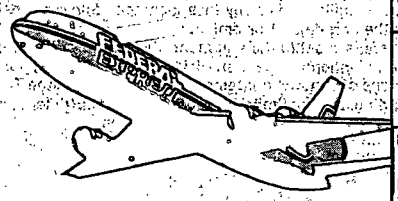
ORM'S AND RADIOACTIVE MATERIAL ONLY

OVERNIGHT IS NEXT BUSINESS DAY (MONDAY THROUGH FRIDAY); TWO DAYS FROM ALASKA/HAWAII. SATURDAY DELIVERY AVAILABLE IN CONTINENTAL U.S. SEE "SPECIAL HANDLING."

RECEIVED AT: ☐ SHIPPER'S DOOR ☐ REGULAR STOP ☐ ON-CALL STOP ☐ F.E.C. LOC.

Federal Express Corporation Employee No.: 32174

DATE/TIME For Federal Express Use: 6 09-53



AIRBILL NUMBER 768844333

SHIPPER'S CERTIFICATION FOR RESTRICTED ARTICLES

(TYPE OR PRINT)

NO. OF PKGS.	PROPER SHIPPING NAME (PER 49 CFR 172.101)	CLASSIFICATION	IDENTIFICATION NO.	ORIGINAL (Rec) NET QUANTITY PER PACKAGE
1	Flammable Solid N.O.S.		UN 1325	1 KGM

ADDITIONAL DESCRIPTION REQUIREMENTS FOR RADIOACTIVE MATERIALS (SEE BACK)

RADIONUCLIDE	FORM	ACTIVITY	CATEGORY OF LABELS	TRANS. INDEX	PACKAGE IDENTIFICATION

THIS SHIPMENT IS WITHIN THE LIMITATIONS PRESCRIBED FOR: PASSENGER AIRCRAFT / CARGO AIRCRAFT ONLY (DELETE-NONAPPLICABLE)

IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS OR TREATMENT.

I HEREBY CERTIFY THAT THE CONTENTS OF THIS CONSIGNMENT ARE FULLY AND ACCURATELY DESCRIBED ABOVE BY PROPER SHIPPING NAME AND ARE CLASSIFIED, PACKED, MARKED, AND LABELED, AND IN PROPER CONDITION FOR CARRIAGE BY AIR ACCORDING TO APPLICABLE NATIONAL GOVERNMENTAL REGULATIONS.

NAME AND TITLE OF PERSON SIGNING CERTIFICATION	EMERGENCY TELEPHONE NO.	SIGNATURE OF SHIPPER
R. J. Slaboda, Chemist, NUS Corp.	215 687 9510	

1. In tendering the shipment for carriage the shipper agrees to these TERMS AND CONDITIONS OF CONTRACT which no agent or employee of the parties may alter and that this Federal Express Airbill is NON-NEGOTIABLE and has been prepared by him/or on his behalf by Federal Express.

2. The shipper agrees that carriage is subject to terms and conditions of contract stated herein and those terms and conditions which are also stated in the most recent Federal Express Service Guide, which is available for inspection and incorporated into this contract by reference.

3. In tendering the shipment for carriage, **THE SHIPPER WARRANTS** that the shipment is packaged adequately to protect the enclosed goods and to insure safe transportation with ordinary care and handling, and that each package is appropriately labeled and is in good order (except as noted) for carriage as specified.

4. When the destination of the shipment is not within the Federal Express air terminal zone as listed in the most recent Federal Express Service Guide, Federal Express makes no commitment with respect to time of delivery of the shipment.

5. In the event of international carriage of any shipment hereunder, the rules relating to liability established by the Convention for the Unification of Certain Rules Relating to International Carriage by Air signed at Warsaw, Poland on October 12, 1929 shall apply to the carriage insofar as the same is governed thereby.

6. Federal Transportation Excise Tax. Pursuant to Section 4271 of the Internal Revenue Code, a 5% tax on air transportation portion of the service and the accessorial services related thereto is included within the basic rate.

7. DECLARED VALUE AND LIMITATION OF LIABILITY. **THE LIABILITY OF FEDERAL EXPRESS IS LIMITED TO THE SUM OF $100.00** unless a higher value is declared for carriage herein and a greater charge paid at the rate of 30¢ per $100.00 value. The maximum higher declared value is $5000.00. Shipments containing items of extraordinary value, including, but not limited to, drawings, paintings, sculptures, porcelain, ceramics, furs, fur clothing, fur trimmed clothing, jewelry, watches, gems, stones (precious or semi-precious, cut or uncut), industrial diamonds, costume jewelry, precious metals, gold, silver, (bullion, dust or precipitates), platinum (except as an integral part of electronic machinery), money, currency, coins, trading stamps, stocks, bonds, cash letters (or their equivalent) or other extraordinary valuable items, are limited to a maximum declared value of $500.00. When multiple packages are placed on a single airbill but the shipper has not specified the declared value of each individual package, the declared value for each individual package will be determined by dividing the total declared value on the airbill by the number of packages indicated on the airbill, subject to a $100.00 minimum declared value per individual package. The liability of Federal Express is limited to the declared value of the shipment or the amount of loss or damage actually sustained, whichever is lower.

Federal Express is not liable for loss, damage, delay, mis-delivery or non-delivery not caused by its own negligence or any loss, damage, delay, mis-delivery or non-delivery caused by the act, default or omission of the shipper, consignee, or any other party who claims interest in the shipment, the nature of the shipment or any defect, characteristic of inherent vice thereof; violation by the shipper or consignee of any of the conditions of contract contained in this airbill or in the Federal Express Service Guide, including, but not limited to, improper or insufficient packing, securing, marking or addressing, or failure to observe any of the rules relating to shipments not acceptable for transportation or shipments acceptable only under certain conditions; acts of God, perils of the air, public enemies, public authorities acting with actual or apparent authority, authority of law, acts or omissions of customs or quarantine officials, riots, strikes or other local disputes, civil commotions, hazards incident to a state of war, weather conditions or mechanical delay of the aircraft or acts or omissions of any person other than FEC, including compliance with delivery instructions from the shipper or consignee. FEC shall not be liable for the loss of articles loaded and sealed in packages by the shipper provided the seal is unbroken at the time of delivery and the package retains its basic integrity. **FEDERAL EXPRESS SHALL NOT BE LIABLE IN ANY EVENT FOR ANY SPECIAL, INCIDENTAL OR CONSEQUENTIAL DAMAGES, INCLUDING BUT NOT LIMITED TO LOSS OF PROFITS OR INCOME WHETHER OR NOT FEDERAL EXPRESS HAD KNOWLEDGE THAT SUCH DAMAGES MIGHT BE INCURRED.**

8. CLAIMS. **WRITTEN NOTICE OF LOSS DUE TO DAMAGE, SHORTAGE OR DELAY MUST BE REPORTED BY THE SHIPPER WITHIN 15 DAYS AFTER THE DELIVERY OF THE SHIPMENT. WRITTEN NOTICE OF LOSS DUE TO NON-DELIVERY MUST BE REPORTED BY THE SHIPPER WITHIN 180 DAYS AFTER ACCEPTANCE OF THE SHIPMENT OR CARRIAGE.** Written notification will be considered to have been made if the shipper calls and notifies the Customer Services Department at 800/238-5355 (in Tennessee 800/542-5171) and as soon as practicable thereafter files a written notification. Documentation of all claims other than overcharge claims must be submitted in writing to FEC within ninety (90) days after receipt of written notification. No claim for damage will be entertained until all transportation charges have been paid. The amount of a claim may not be deducted from the transportation charges. Receipt of the shipment by the consignee without written notification of damage on the delivery receipt shall be prima facie evidence that the shipment was delivered in good condition, except that in the case of claims for concealed damage which is not discovered at the time of delivery, the shipper shall notify FEC in writing as promptly as possible after the discovery thereof and in any event not later than 15 days from the date of delivery. The shipper must make the original shipping cartons and packing available for inspection by FEC. Claims for overcharges and refunds must be made in writing to FEC within twelve (12) months of the billing date. All claims must be filed by the shipper.

9. All shipments are subject to inspection by FEC, including but not limited to, opening the shipment. However, FEC is not obligated to perform such inspection.

10. C.O.D. services are not available and a C.O.D. shipment sent in error will be delivered as a normal pre-paid or collect shipment.

11. Federal Express carries no cargo liability insurance but maintains a separate fund for the satisfaction of cargo claims which may arise out of the carriage of cargo pursuant to the conditions of contract contained herein and in the most recent Federal Express Service Guide.

12. Notwithstanding the shipper's instructions to the contrary, the shipper shall be primarily liable for all costs and expenses related to the shipment of the package, and for costs incurred in either returning the shipment to the shipper or warehousing the shipment pending disposition.

13. Saturday Delivery: Recipient's phone number is required.

7688 4233

RADIOACTIVE MATERIAL SHIPMENT INFORMATION

Radionuclide	-	Element and mass number
Form	-	Special form or chemical and physical form
Activity	-	Use appropriate units - Ci -curie, mCi - millicurie, uCi - microcurie
Label	-	White I, Yellow II, Yellow III, or None when no label required.
Transport Index	-	For Yellow II and Yellow III labeled packages only
Package Identification	-	NRC Certificate of Compliance identification number; or Certificate of Competent Authority identification number; or package type, if applicable, and not included in identification number; or package specification if none of above not applicable.

WARNING: Failure to comply in all respects with the applicable regulations of the Department of Transportation, 49 CFR, Parts 100-199 and, for international shipments, the IATA Restricted Articles Regulations may be a breach of the applicable law, subject to legal penalties. This certification shall in no circumstance be signed by an IATA Cargo Agent or a consolidator for international shipments.

ORIGINAL
(Red)

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number
619C

SPECIAL ANALYTICAL SERVICE
PACKING LIST

Sampling Office: III

Sampling Contact: T. Shannon (name)

215-687-9510 (phone)

Sampling Date(s): 6/21/83

Date Shipped: 6/29/83

Site Name/Code: m-02

Ship To: Envirodyne
12161 Lakeland Rd.
St. Louis, Mo. 63141

Attn: Judy Stone

For Lab Use Only

Date Samples Rec'd:

Received By:

	Sample Numbers	Sample Description i.e., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1.	m-02-01	2,3,7,8-TCDD Analysis / Solid / Low	
2.	m-02-02	2,3,7,8-TCDD Analysis / Solid / Low	
3.	m-02-03	2,3,7,8-TCDD Analysis / Solid / Low	
4.	m-02-04	2,3,7,8-TCDD Analysis / Solid / Low	
5.	m-02-05	2,3,7,8-TCDD Analysis / Solid / low	
6.	m-02-06	2,3,7,8-TCDD Analysis / Solid / Low	
7.	m-02-07	2,3,7,8-TCDD Analysis / Solid / Low	
8.	m-02-08	2,3,7,8-TCDD Analysis / ~~Solid~~ Aqueous (TAS) / Low	
9.	m-02-09	2,3,7,8-TCDD Analysis / Solid / Low	
10.	m-02-10	2,3,7,8-TCDD Analysis / Solid / Low	
11.	m-02-12	2,3,7,8-TCDD Analysis / Solid / low	
12.	m-02-13	2,3,7,8-TCDD Analysis / Solid / Low	
13.	m-02-14	2,3,7,8-TCDD Analysis / Solid / Low	
14.	m-02-14	2,3,7,8-TCDD Analysis / Solid / Low / "To Be Spiked By Laboratory"	
15.			
16.	m-01-01	2,3,7,8-TCDD Analysis / ~~Aqueous~~ Solid (TAS) / High	
17.		Note: Sample is hexachlorophene powder.	
18.	m-02-15	2,3,7,8-TCDD Analysis / Solid / Low	
19.	m-02-16	2,3,7,8-TCDD Analysis / Solid / Low	
20.			

For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

ORIGINAL
(Red)

On June 21, 1983, C.A. Shaheen gave permission for Terrance B. Shannon, of NUS Corp. to remove property from their facility in the following containers: 15- one pint jars for 2,3,7,8- TCDD analysis, and 24- eight ounce jars.

C.A. Shaheen 6/21/83
Facility Representative Date

Terrance B. Shannon 6/21/83
NUS Representative Date

ORIGINAL
(Red)

Appendix F

Lab: Battelle

Case:

Shipment:

Date: 10/19/83

GC Column: CP Sil-88

Average Native RRF: 0.95

Average Surrogate RRF: 1.24

Sample Number	Extraction Method	Cleanup Option	Gm. Wt.	PPB TCDD	D.L.	Analytical Date	Analytical Time	Native m/z 320 / m/z 322 Area Ratio	C-13 m/z 332 / m/z 334 Area Ratio	Surrogate (1) Percent Accuracy	m/z 320 area	m/z 322 area	m/z 257 area	m/z 328 area	m/z 332 area	m/z 334 area	Comments
13228	J	B	10.8			10/19				UNABLE TO ANALYZE DUE TO PRECIPITATE							
M-02-13	J	B	10.8			10/19				"							
M-02-14	J	B	10.9	ND	.02	10/19	--	.95	.80	120	31.90	33.53	--	7711.42	7091.71	8917.86	
M-02-16	J	B	10.4	3.47	--	10/19	--	.78	.78	121	8950.35	11533.43	--	7235.18	6499.87	8283.64	
M-02-14N	J	B	10.7	1.58	--	10/19	--	.78	.80	119	4819.68	6141.26	--	8358.66	7767.68	9703.96	
MB	J	B	10.0	0.01	--	10/19	--	.77	.81	119	27.01	35.16	--	7026.23	6582.39	8158.30	
M-02-13	J	B	1.0	ND	.83	10/19	--	.17	.76	120	11.78	68.82	--	3049.33	2798.36	3544.60	

(1) Corrected for contribution by native TCDD (Subtract 0.009 of m/z 322).
(2) Based on 10 gram sample.

MB = Method Blank
P = Partial Scan
N = Native TCDD Spike
D = Duplicate (Intralab)
FB = Field Blank

H = High Resolution
ND = Not Detected
DL = Detection Limit
J = Jar Extraction

A,B,C = Cleanup Option (or any combination)

ORIGINAL (Red)

ORIGINAL
(Red)

EXHIBIT II -COMPOUND LIST

SAMPLE IDENTIFIER: #13
COMPUCHEM SAMPLE NUMBER: 13228 2nd analysis

COMPOUND	RESULT (ppb)	DETECTION LIMIT (ppb)
2378-TCDD	Not Detected	0.20

ORIGINAL
(Red)

EXHIBIT II -COMPOUND LIST

SAMPLE IDENTIFIER: #13
COMPUCHEM SAMPLE NUMBER: 13228 1st analysis

COMPOUND	RESULT (ppb)	DETECTION LIMIT (ppb)
2378-TCDD	Not Detected	0.70

TABLE 1 - DATA SUMMARY

Lab: Envirodyne Engineers, Inc.

Date: 9/20/83

Case: 619C Rerun

GC Column: SP-2330, 60 meter

Sample Number	Extraction	Cleanup	Wet Weight (grams)	TCDD (ug/kg)	D.L. (ug/kg)	Analytical Date	Analytical Time	320/322	332/334	Surrogate Percent Accuracy	257	320	322	328[a]	332	334	FRN Numbers
619C-M02-13	J	A,D	10.4	1.0	-	8/26	0040	0.67	0.86	80	58	87	130	250	280	328	7384

Notes: MB = Method Blank
J = Jar Extraction
A,B,C,D = Cleanup Option from Method
FRN = File Reference Number
D = Duplicat
D.L. = Detection Limit
N = Native 2,3,7,8-TCDD Spike

[a]Corrected for contribution by native TCDD (subtract 0.009 of m/e 322).

ORIGINAL (Red)

TABLE 1

DATA SUMMARY

Lab: Envirodyne Engineers, Inc.

Date: July 22, 1983

Case: 619C

GC Column[a]: SP-2330, 60 meters
SP-2340, 60 meters

Sample Number	Extraction	Cleanup	Wet Weight (grams)	TCDD (ppb)	D.L. (ug/kg)	320/322	Surrogate Percent Recovery	257	320	322	328[b]	332	334	FRN[c] Number
M-02-01	J	A,D	10.5	-	0.63	-	111	-	-	-	414	346	474	23601
M-02-02	J	A	10.3	-	0.22	-	93	-	-	-	999	1106	1349	23525
M-02-03	J	A	10.1	-	0.20	-	83	-	-	-	678	843	1032	23526
M-02-04	J	A,D	10.3	-	0.21	-	102	-	-	-	741	742	922	23624
M-02-04D	J	A,D	10.1	-	0.14	-	101	-	-	-	629	662	767	23625
M-02-05	J	A	10.1	-	0.06	-	85	-	-	-	1306	1550	1955	23529
M-02-06	J	A,D	10.8	-	0.15	-	95	-	-	-	1512	1564	1919	23602
M-02-07	J	A,D	10.6	-	0.19	-	91	-	-	-	528	591	738	23623
M-02-09	J	A	11.1	-	0.47	-	92	-	-	-	810	902	1110	23533
M-02-10	J	A,D	10.4	-	0.14	-	98	-	-	-	1698	1698	2116	23604
M-02-12	J	A,D	10.7	-	0.69	-	93	-	-	-	193	203	272	23620
M-02-13	J	A,D	10.4	-	Interferences, is being re-extracted and reanalyzed; See FRNs 23536 and 23621.									
M-02-14	J	A,D	10.8	-	0.46	-	104	-	-	-	111	99	145	23622
M-02-14N	J	A	10.1	1.2	-	0.73	93	142	352	481	895	999	1208	23539[d]
M-02-14N[a]	J	A,D	10.1	1.3	-	0.82	104	462	1103	1340	2618	4263	5330	6912
M-02-15	J	A	10.1	3.3	-	0.82	94	336	723	880	639	706	845	23541[d]
M-02-15[a]	J	A	10.1	3.6	-	0.74	113	731	2376	3226	2042	3290	4340	6911[d]
M-02-16	J	A	10.1	3.1	-	0.79	95	682	1529	1933	1452	1556	1969	23542
M-02-16[a]	J	A	10.1	3.4	-	0.78	113	839	2881	3677	2531	4201	5271	6913
MB (soils)	J	A	-	-	0.24[e]	-	83	-	-	-	646	797	989	23545
M-01-01 (powder)	J	A,B	10.1	-	0.20	-	99	-	-	-	308	329	387	23547
MB (powder)	J	A,B	-	-	0.84[e]	-	93	-	-	-	85	95	114	23548
M-02-08 (rinsate)	-	A	65[f]	-	0.039[g]	-	95	-	-	-	1297	1436	1697	23532

ORIGINAL (Red)

TABLE 1

DATA SUMMARY

Lab: Envirodyne Engineers, Inc.

Date: July 22, 1983

Case: 619C

GC Column[a]: SP-2330, 60 meters
SP-2340, 60 meters

Sample Number	Extraction	Cleanup	Wet Weight (grams)	TCDD (ppb)	D.L. (ug/kg)	320/322	Surrogate Percent Recovery	257	320	322	328[b]	332	334	FRN[c] Number

Notes: MB = Method Blank
J = Jar Extraction
A,B,C = Cleanup Option from Method
FRN = File Refence Number
D = Duplicate
D.L. = Detection Limit
N = Native 2,3,7,8-TCDD Spike

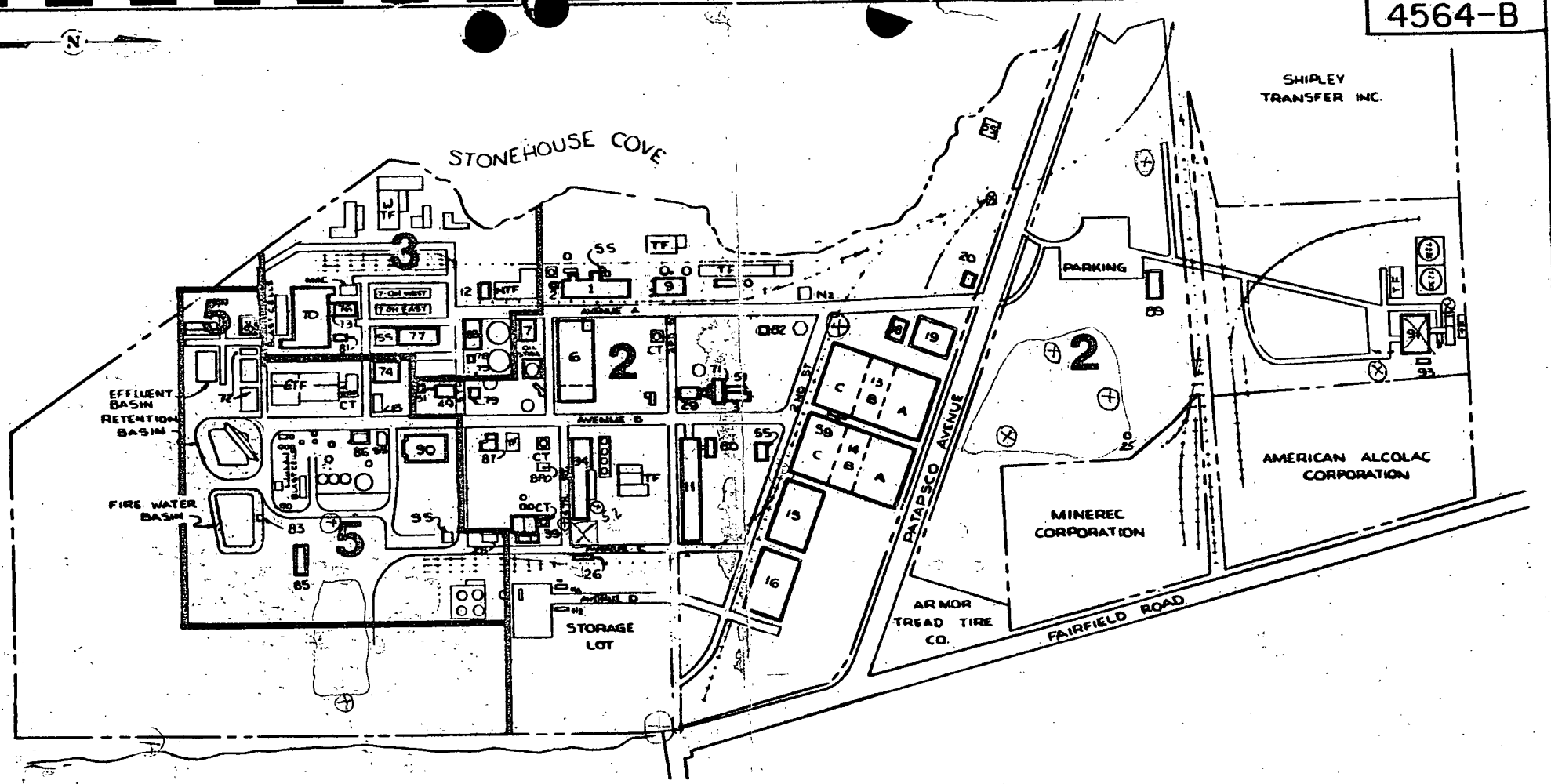
[a]Samples were all initially analyzed on SP-2340 column. Positive values were confirmed on the SP-2330 column.
[b]Corrected for contribution by native TCDD (subtracted 0.009 of m/e 322).
[c]Samples with FRNs 23xxx analyzed on HP-5985 (SP-2340 column). Samples with FRNs 69xx were analyzed on HP-5993 (SP-2330 column).
[d]Samples run just prior to these positives:

Positive Sample	FRN	Previous Sample	FRN
M-02-14N	23539	M-02-14	23538
M-02-15	23541	Mixed Isomer standard	23540
M-02-15 confirmation	6911	Another project	6910

[e]Assumes 10 g sample.
[f]In milliliters
[g]In ug/l.

ORIGINAL (Red)

4564-B



BUILDINGS

1 POWER HOUSE
2 STACK
6 ETHION & BUTOXIDE BLDG.
7 P_2S_5 CONVEYOR & STORAGE BLDG.
9 DIALLYL PHTHALATE BLDG.
10 PACKAGING BLDG.
11 CENTRAL SHOP & STOREROOM
12 FIRE PUMP HOUSE
13-A WAREHOUSE
13-B CARPENTER SHOP
13-C WAREHOUSE & SHIPPING DEPT.
14-A STORAGE
14 B STORAGE
14-C DRUM RECOVERY
15 WAREHOUSE
16 WAREHOUSE
18 MISC. STORAGE
19 MAIN OFFICE BLDG.
20 GATE HOUSE
25 BOOSTER PUMP HOUSE
26 ANHYDROUS AMMONIA
28 HCl LOADING BLDG.
29 CONTROL LAB
31 PPODUCTION OFFICE
34 DV ESTER
39 LOAD CENTER
44 STORAGE
45 STORAGE
49 SODIUM ALKYLATES
51 SODIUM STORAGE
54 LOCKERS
55 CAFETERIA
59 PAINT STORAGE
70 7-OH PLANT II
71 CONTROL LAB
72 INCINERATOR AREA
73 7-OH CONTROL ROOM
74 7-OH STORE ROOM
75 EFFLUENT CONTROL ROOM
76 #2-7-OH CONTROL & LOCKER ROOMS
77 7-OH SHOP
78 CL_2 CONTROL ROOM
79 FIRE PUMP HOUSE #1
80 LOCKERS
81 REFRIGERATOR BLDG.
82 LOCKERS
83 FIRE PUMP HOUSE #2
84 7-OH DAY CHEMICAL BLDG.
85 PLANT III, 7-OH SHOP & WAREHOUSE
86 MCC, #3-7-OH CONTROL & LOCKER ROOM
87 LOCKERS
88 CALGON CARBON BLDG.
89 POUNCE STORAGE
90 OPERATIONS BLDG.
91 POUNCE PLANT
93 ELEC. CONTROL ROOM
CT COOLING TOWER
TF TANK FARM
SS SUB STATION
CS COMPRESSOR BLDG.

AREAS

AREA 2 ACTIVE - SHOP, OFFICES, BUILDINGS 1, 4, 7, 9, 91 LAB, WAREHOUSES & HYDROGEN STATION

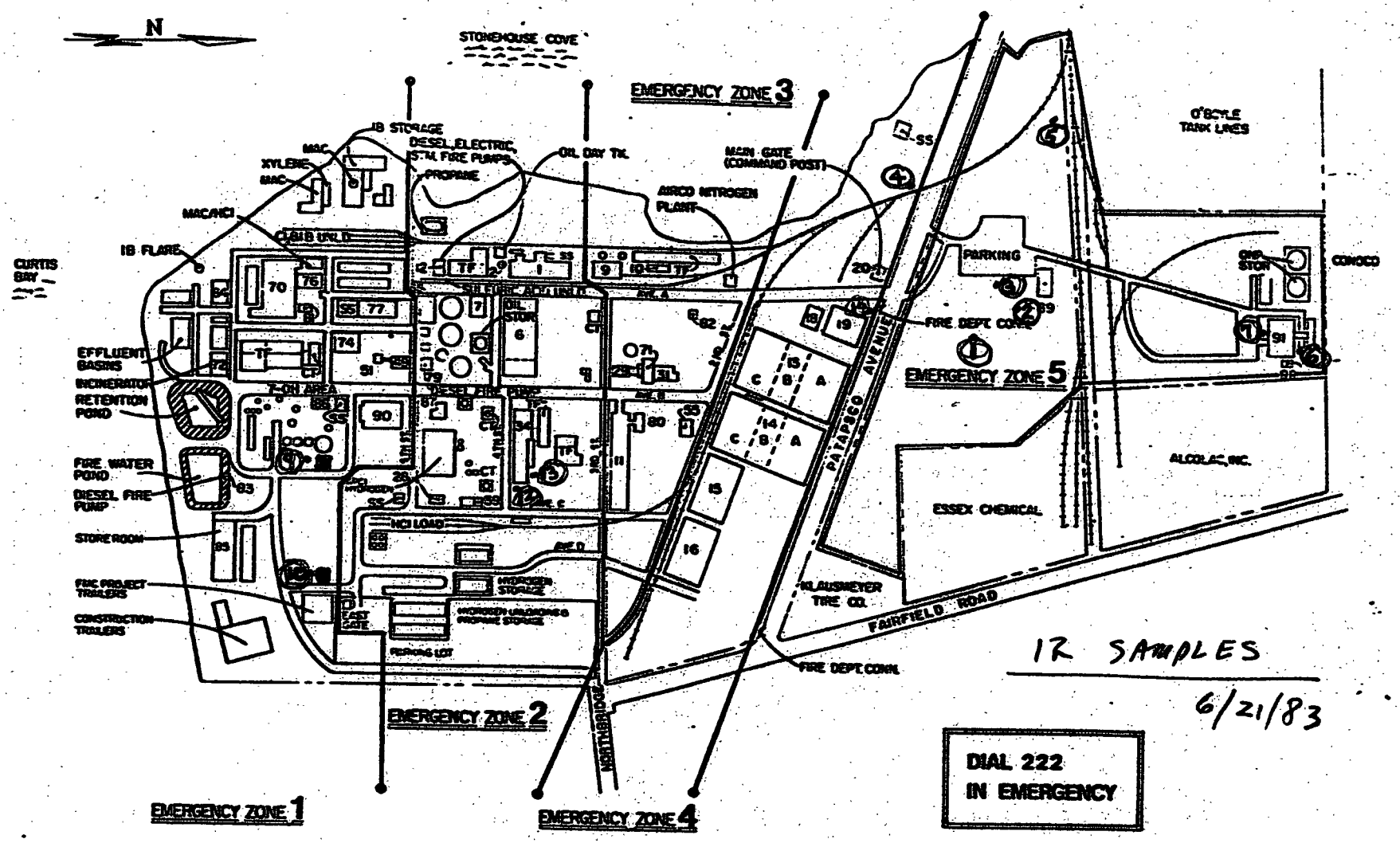
AREA 3 ACTIVE - NORTH & WEST TANK FARMS, 7-OH PLANT I, MAC & HCl PLANTS PLANT II CONTROL ROOM, STEPS IIA, IIB2, III, IV ETC.

AREA 5 ACTIVE - 7-OH PLANT III, PLANT III CONTROL ROOM, WASTE BASINS, HCl STORAGE, INCINERATOR COOLING TOWERS & EAST TANK FARM ETC.

MAINTENANCE AREAS FMC-BALTIMORE PLANT

ORIGINAL (1)

REV. 4/5/82



No. 8
No. 11
} No Samples

ORIGINAL (Red)

		9	ADD LP TO ZONE 2	GAB	drawn by: RW BROWN
					approved by:
					JULY 30, 1982

REVISIONS

4564-b | rev. no. 2